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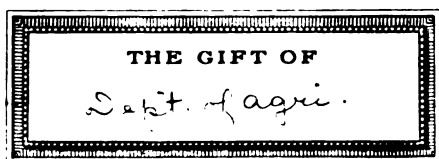
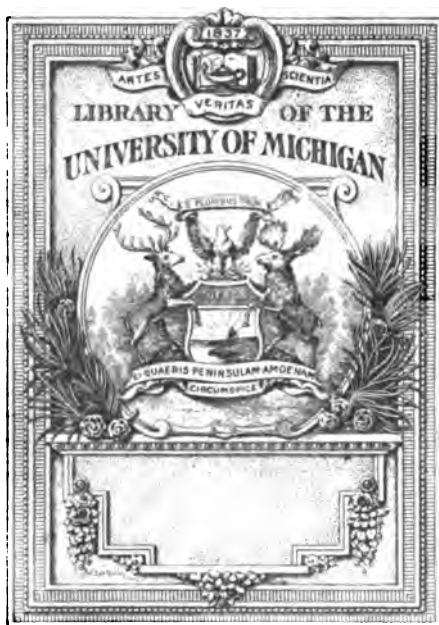
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Issued October 27, 1908.

U. S. DEPARTMENT OF AGRICULTURE.

BUREAU OF CHEMISTRY—BULLETIN No. 116.

H. W. WILEY, Chief of Bureau.

PROCEEDINGS

OF THE

TWENTY-FOURTH ANNUAL CONVENTION

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS,

HELD AT

THE JAMESTOWN EXPOSITION, NORFOLK, VA.,
OCTOBER 9-11, 1907.

EDITED BY

HARVEY W. WILEY,
SECRETARY OF THE ASSOCIATION.



WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1908.

LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., May 18, 1908.

SIR: I have the honor to submit for your approval the Proceedings of the Twenty-fourth Annual Convention of the Association of Official Agricultural Chemists. All addresses and discussions have been briefly summarized and the reports submitted in the most concise form practicable. I recommend that these proceedings be published as Bulletin No. 116 of the Bureau of Chemistry.

Respectfully,

H. W. WILEY,
Chief of Bureau.

HON. JAMES WILSON,
Secretary of Agriculture.

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PROCEEDINGS OF THE TWENTY-FOURTH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

FIRST DAY.

WEDNESDAY—MORNING SESSION.

The twenty-fourth annual convention of the Association of Official Agricultural Chemists was called to order by the president, Mr. J. P. Street, of New Haven, Conn., on the morning of October 9, in the assembly hall of the Inside Inn, Jamestown Exposition, Norfolk, Va.

The following members and visitors registered during the convention:

MEMBERS AND VISITORS PRESENT.

Allen, William M., Department of Agriculture, Raleigh, N. C.
Atkinson, Frederick C., District Chemist, Southern Cotton Oil Company, Savannah, Ga.
Averitt, S. D., Agricultural Experiment Station, Lexington, Ky.
Bailey, Herbert S., Bureau of Chemistry, Washington, D. C.
Bartlett, J. M., Agricultural Experiment Station, Orono, Me.
Birdsey, C. E., New York, N. Y.
Bizzell, James A., 804 E. Seneca street, Ithaca, N. Y.
Blair, A. W., Agricultural Experiment Station, Gainesville, Fla.
Browne, Charles A., Bureau of Chemistry, Washington, D. C.
Brinton, Clement S., U. S. Appraiser's Stores, Philadelphia, Pa.
Bryan, Thomas J., Illinois State Food Commission, Manhattan Building, Chicago, Ill.
Burnet, Wallace C., Bureau of Chemistry, Washington, D. C.
Carpenter, F. B., Virginia-Carolina Chemical Company, Richmond, Va.
Carroll, John S., German Kali Works, Atlanta, Ga.
Cathcart, Charles S., Agricultural Experiment Station, New Brunswick, N. J.
Cook, Frank C., Bureau of Chemistry, Washington, D. C.
Cook, H. H., Waterbury, Conn.
Curry, B. E., Durham, N. C.
Davidson, Robert J., Agricultural Experiment Station, Blacksburg, Va.
Dodge, C. O., Washington, D. C.
Doyle, Aida M., 1025 Lamont street, Washington, D. C.
Dox, Arthur W., Assistant Dairymen, Bureau of Animal Industry, Storrs, Conn.
Edmonds, H. D., Storrs, Conn.
Ellett, Walter B., Agricultural Experiment Station, Blacksburg, Va.
Fraps, G. S., Agricultural Experiment Station, College Station, Tex.

Frear, William, Agricultural Experiment Station, State College, Pa.
 Fuller, F. D., Department of Agriculture, Harrisburg, Pa.
 Fuller, Mrs. F. D., Harrisburg, Pa.
 Gaither, E. W., Nashville, Tenn.
 Gearkens, A., Antwerp, Belgium.
 Gudeman, Edward, Consulting Chemist, 704 Rialto Building, Chicago, Ill.
 Hand, William F., Agricultural Experiment Station, Agricultural College, Miss.
 Hanson, Herman H., Agricultural Experiment Station, Orono, Me.
 Hartwell, Burt L., Agricultural Experiment Station, Kingston, R. I.
 Haskins, H. D., Amherst, Mass.
 Haywood, William G., Department of Agriculture, Raleigh, N. C.
 Heide, Henry, 313 Hudson street, New York, N. Y.
 Herff, B. von, 93 Nassau street, New York, N. Y.
 Hite, B. H., Agricultural Experiment Station, Morgantown, W. Va.
 Hopkins, Cyril G., University of Illinois, Urbana, Ill.
 Horne, W. D., National Sugar Refining Company, Yonkers, N. Y.
 Houghton, Harry W., Bureau of Chemistry, Washington, D. C.
 Howard, Burton J., Bureau of Chemistry, Washington, D. C.
 Jackson, H. C., Bender Hygienic Laboratory, Albany, N. Y.
 Kebler, Lyman F., Bureau of Chemistry, Washington, D. C.
 Kilgore, B. W., State Chemist, Raleigh, N. C.
 Lipman, Jacob G., Agricultural Experiment Station, New Brunswick, N. J.
 Lowenstein, Arthur, Morris & Co., Chicago, Ill.
 McDonnell, H. B., Maryland Agricultural College, College Park, Md.
 McGill, Anthony, Chemist to Canadian Government, Ottawa, Canada.
 Mason, Glen F., H. J. Heinz Company, Pittsburg, Pa.
 Michael, Louis G., Agricultural Experiment Station, Ames, Iowa.
 Moore, C. C., Department of Agriculture, Washington, D. C.
 Morrison, William G., F. S. Royster Guano Company, Norfolk, Va.
 Patrick, G. E., Bureau of Chemistry, Washington, D. C.
 Patten, Andrew J., Agricultural Experiment Station, Agricultural College, Mich.
 Patterson, H. J., Agricultural Experiment Station, College Park, Md.
 Pierce, Anne L., Bureau of Chemistry, Washington, D. C.
 Pinkerton, Thomas C., Walbrook, Baltimore, Md.
 Porch, Madison B., Hygienic Laboratory, Washington, D. C.
 Richardson, W. D., Swift & Co., Chicago, Ill.
 Richardson, Mrs. W. D., Chicago, Ill.
 Robb, John B., Department of Agriculture, Richmond, Va.
 Robertson, B. F., Agricultural Experiment Station, Clemson College, S. C.
 Rose, R. E., Tallahassee, Fla.
 Ross, B. B., State Chemist, Auburn, Ala.
 Rudnick, Paul, Armour & Co., Chicago, Ill.
 Schmitt, Walter, Armour & Co., Chicago, Ill.
 Shedd, O. M., Agricultural Experiment Station, Lexington, Ky.
 Sherwood, Sidney F., Bureau of Chemistry, Washington, D. C.
 Shutt, Frank T., Experiment Farm, Ottawa, Canada.
 Sindall, Harry E., 133 North Front street, Philadelphia, Pa.
 Smith, Frank M., H. J. Baker and Brother, 100 William street, New York, N. Y.
 Smither, F. W., Bureau of Chemistry, Washington, D. C.
 Snyder, Harry, Agricultural Experiment Station, St. Anthony Park, Minn.
 Street, John Phillips, Agricultural Experiment Station, New Haven, Conn.
 Taber, Walter C., Bureau of Soils, Washington, D. C.
 Tolman, L. M., Bureau of Chemistry, Washington, D. C.
 Trowbridge, P. F., Agricultural Experiment Station, Columbia, Mo.

Van Slyke, L. L., Agricultural Experiment Station, Geneva, N. Y.
 Walker, Percy, Bureau of Chemistry, Washington, D. C.
 Warner, H. J., Department of Agriculture, Washington, D. C.
 Wiley, H. W., Bureau of Chemistry, Washington, D. C.
 Wiley, S. W., Wiley & Hoffman, Baltimore, Md.
 Willard, Julius F., Agricultural Experiment Station, Manhattan, Kans.
 Winton, A. L., Food and Drug Inspection Laboratory, Manhattan Building,
 Chicago, Ill.
 Withers, W. A., Agricultural Experiment Station, West Raleigh, N. C.
 Zerban, Fritz, Agricultural Experiment Station, Audubon Park, New Orleans, La.

A communication was presented from the Fertilizer Manufacturers' Association inviting the members of the Association of Official Agricultural Chemists to be their guests at dinner on the evening of October 9, and the secretary was instructed to accept the invitation on behalf of the association.

Letters from Atlantic City, N. J., Asbury Park, N. J., and Columbus, Ohio, inviting the association to hold the meeting of 1908 in the several localities named were also submitted and referred to the executive committee, the secretary being authorized to acknowledge the same.

FOOD ADULTERATION.

In the absence of Mr. Leach, referee on food adulteration, no general report on the subject was made, and the associate referees on the subject presented the subreports in the following order:

REPORT ON COLORS.

By E. F. LADD, *Associate Referee*.

The referee on colors submitted two subreports by Messrs. Hortvet and Gudeman and made the following recommendations, which were presented in his behalf by the secretary:

It is recommended—

(1) That more attention be given to the use of color standards in color work on foods by following the outline and method prepared by Mulliken in "A Method for Identification of Pure Organic Compounds," as suggested by Mr. Julius Hortvet.

(2) That a more comprehensive examination of the so-called "vegetable colors," some of which it would seem are not more properly to be classed as vegetable colors than are the anilin colors, be made during the coming year.

The paper submitted by Mr. Hortvet, entitled "An examination of colors used in foods or natural to foods," is to be found in the Eleventh Biennial Report of the Minnesota State Dairy and Food Commission, pages 583 to 593.

The second subreport, entitled "Solubilities and extraction values of food colors," by Edward Gudeman, is to be found in the Journal of the American Chemical Society, November, 1907, volume 29, No. 11, page 1629. The report contains two tables giving the solubility and extraction values obtained on three vegetable colors and three coal-tar colors of similar shades and possessing exceptional color intensities. The following extract is made, giving the conclusions reached:

Examination of these tables shows that the use of miscible solvents like methyl and ethyl alcohols and acetone are of no value for extraction purposes. Solvents of an acid-like character (ethyl acetate) are not suitable, as the results obtained are misleading, due to the effect of the solvent on the color.

The solubilities of many colors in petroleum ether, carbon disulphid, carbon tetrachlorid, and chloroform is so slight that these solvents are very suitable for preliminary extraction of food products, thereby extracting oils and fats before making dyeing tests to separate the colors. Care must be exercised, as many colors are soluble in fats or oils and are liable to be extracted in such preliminary treatment. Examination of the fats or oils will show whether any color has been extracted with these.

Conclusions drawn from a very large number of solubility and extraction tests, extending over a long period of time, are that the colors extracted or dissolved by many solvents under varying conditions from neutral, acid, or alkaline solutions, give no conclusive data for deciding upon the character or class of the colors themselves. The differences in solubility and in extractive values of vegetable colors, compared with coal-tar colors, are no greater nor less than the differences found between the various colors themselves belonging to the same class of colors.

Comparative color intensities were also determined, and it was found that only a very limited number of vegetable colors had a color intensity equal to one-fourth that of a corresponding shade of coal-tar color, and that the largest number of vegetable colors had one-tenth or less color intensity than the corresponding coal-tar color of similar shade.

In the absence of the referee on saccharine products, the following note from him was presented by the secretary:

REPORT ON SACCHARINE PRODUCTS.

By C. H. JONES, *Associate Referee.*

The referee on saccharine products attempted no cooperative work this year, but has personally examined many representative samples of maple sirup and sugar from the large crop of 1907 and found them to conform to the standards already established. It would seem that with the ash test, and the determination of the lead precipitate, and the malic acid value all ordinary maple substitutes might be readily differentiated from the pure article. A few changes in the determination of the malic acid value, based on the cooperative work done in 1906, have been referred to Mr. J. K. Haywood, chairman of the committee on the revision of methods, for insertion in the methods to be adopted at this convention.

REPORT ON COCOA AND COCOA PRODUCTS.

E. MONROE BAILEY, *Associate Referee.*

The associate referee on cocoa and cocoa products submitted the following methods of analysis by Winton, with the statement that with a few modifications they were the same as those employed at the Connecticut Agricultural Experiment Station, and published in the station report for 1902-3. He further suggested that the method used in the U. S. Department of Agriculture, Bureau of Chemistry (Dubois's method^a), be substituted for section 13, "Sugar," and recommended that these methods be adopted by the association as provisional until the referee could make a fuller report upon this subject.

The methods, with the change in section 13 as suggested, are given in Bulletin 107, Revised, Methods of Analysis, page 254.

DISCUSSION ON SPICES.

The associate referee on spices, A. L. Winton, made no formal report, but stated that the methods for the examination of spices used at present seemed to him fairly satisfactory, and that the efforts of the association might therefore be better devoted to subjects of more pressing need.

Mr. Winton further said:

It has been my privilege to examine numerous shipments of spices in the warehouses of importers, where I have had an opportunity to note not only the general character of the products, but also the extent to which they have been damaged by insects. The nutmegs, known in the trade as grinding nutmegs, have been visited before shipment by some insect which, curiously enough, devours almost completely the starchy portion of the nut, but avoids the resinous veins. As a consequence, the nuts are light in weight and may be easily crushed between the fingers. Such nutmegs grind more easily than the sound product and, owing to the removal of the starchy matter, may contain a higher percentage of volatile oil; they are, however, unfit for consumption and their sale is a direct violation of the food and drugs act, on the ground that they are in whole or in part a filthy, decomposed, or putrid vegetable substance.

Whole ginger during long storage is almost sure to be attacked by the drug-store beetle, which makes numerous burrows through the hands and finally converts the product almost entirely into a disgusting powder.

Cayenne pepper and paprika, both before and after grinding, suffer severely from the attacks of a moth, the larva of which bores through the material and finally is transformed into the adult stage with the formation of a web. On opening a bottle of cayenne pepper which has been closed for months or even years a number of these moths will often fly out of the bottle.

Mr. KEBLER. Considerable quantities of worm-eaten ginger are imported into the United States. Several samples were detained at the ports, and at the hearing it developed that the worm-eaten ginger was ground and used in the manu-

^a J. Amer. Chem. Soc., 1907, 29: 556; U. S. Dept. Agr., Bureau of Chemistry, Bul. 107, Rev., Appendix, page 256.

facture of ginger cookies and similar products. Most of the so-called Calcutta ginger is of inferior quality, being usually mixed with abnormal quantities of dirt, the ginger itself never having been cleaned.

A few days ago I visited a large drug warehouse and inspected a number of deliveries of ginger, and in no case was there any material amount worm-eaten. Some of the Jamaica ginger had been slightly attacked by insects, but a large lot of African ginger which had been in the warehouse over four years did not show the slightest indication of such deterioration.

Mr. MCGILL. Is oil of nutmeg an article of commerce? I have not happened to find it on the Canadian market. I can conceive that nutmegs might even be increased in value for purposes of oil extraction by the removal of nonstarch matters by worms or otherwise. There are probably in attendance some persons who can give first-hand information as regards this question.

Mr. KEBLER. Not only is a considerable amount of nutmeg oil itself used, but the oil obtained from the mace is also employed in the manufacture of certain commodities.

FLAVORING EXTRACTS.

No report was submitted by the associate referee on flavoring extracts. The following recommendation in regard to the work for next year on this subject was offered by C. S. Brinton:

In view of the requirements of the new food and drugs act, and the standards adopted by the Department of Agriculture, it is necessary that we should have more definite information on this subject, especially on vanilla extracts. The standards require the soluble matter of 10 grams of vanilla beans in each 100 cc of finished extract. I therefore recommend that the associate referee on this subject for next year be instructed to investigate, and, if possible, to devise new methods for the detection of caramel, and also to investigate the composition and quantity of the soluble matters obtained from vanilla beans in preparing vanilla extract.

The associate referee on cereal products, A. McGill, of Ottawa, Canada, reported that the research work he had undertaken early in the year was not yet completed, because of the new duties which he had assumed upon the death of the chief analyst of Canada, the direction of the laboratories necessitating that the investigations referred to be laid aside.

REPORT ON PRESERVATIVES.

By W. D. BIGELOW, *Associate Referee.*

SALICYLIC ACID.

During the last year a question has arisen as to the applicability of the method commonly used for the detection of salicylic acid in dark colored malt liquors and other products colored dark brown by heat. It has been claimed by manufacturers that errors have occurred because of the similarity of the color reaction with ferric chlorid and with salicylic acid. Under the circumstances it seemed advisable to investigate carefully the application of the methods for the detection of salicylic acid to such products.

Samples were obtained of beer and other malt liquors manufactured from highly colored malt and of breakfast foods colored by heating to a high tem-

perature. These samples were examined independently by A. M. Doyle and P. B. Dunbar of the Bureau of Chemistry, neither of whom had any difficulty in detecting added salicylic acid in any of the products.

It was found that on the addition of a ferric chlorid solution directly to the residue in the dish in which the ether extract was evaporated a dark red or purplish color was sometimes formed. If the dried ether residue was first dissolved in water, however, and the ferric chlorid added to the solution, no difficulty was experienced in any case. Very small amounts of salicylic acid were sometimes obscured by the color of the solution, but no color was obtained in the absence of salicylic acid which would lead the analyst to suspect its presence. A positive reaction for salicylic acid may be confirmed by the Millon reagent, which gives a light pink reaction in the absence of salicylic acid and a deep red in its presence. The ferric chlorid reaction was regarded by both observers as superior in such products to that of the Millon reagent.

FORMALDEHYDE.

The influence of the strength of the hydrochloric acid employed in the hydrochloric acid—ferric chlorid reagent suggested by Leach has been studied during the last year by A. V. H. Mory. It is known that the purple color given by proteids when heated with strong hydrochloric acid is a disturbing factor in the detection of formaldehyde in milk, and various methods have been adopted to avoid error or uncertainty. Some analysts have been accustomed to dilute the hydrochloric acid considerably, others to maintain a temperature sufficiently low to prevent the formation of a proteid reaction, and still others to guard against the difficulty by limiting the time of heating. In order to determine whether such dilution of the acid as would make the proportion of acid to milk a negligible quantity could be made without seriously interfering with the delicacy of the test for formaldehyde, the following dilutions of acid, each containing the specified amount of ferric chlorid, were heated to the boiling point with formaldehyde-free milk, in the proportion of two parts of the acid reagent to one of milk. This temperature and ratio were used because it is very easy to overheat, and the quantities used are generally not measured; also because excess of heat and of acid are known to be the chief sources of trouble.

Hydrochloric acid (sp. gr. 1.19).	Water.
cc.	cc.
100	10
90	20
80	30
70	40
60	

These mixtures were heated with stirring over a free flame in a casserole. The strongest acid gave a deep purple color, which began to develop below 70° C., and deepened quickly to a dark purple. The weakest acid gave practically no color even on boiling and after standing, while the intermediate strengths gave colors increasing regularly in depth and in purple tint with increase in acid strength.

The same experiment, except that milk containing a small quantity of formaldehyde (1 in 250,000) was used, showed a marked falling off in the delicacy of the test with dilution of the acid.

The general results of other experiments along this line show that if equal (measured) quantities of strong acid (sp. gr. 1.19) and milk be used and the

heating not carried beyond 90° C., little or no color is given by formaldehyde-free milk; but if, on the other hand, the acid is in excess or the heating is carried too near the boiling point, the protoid reaction becomes a factor increasing with the excess of acid and with the temperature.

Applying these results to the method referred to, it would appear to be desirable to replace the expression "about" [line 1 (e), p. 185, Bul. 107, Rev.] with the specification that equal (measured) quantities of the strong acid reagent and milk must be used, and to warn the analyst against the excessive use of either acid or heat, since either may produce a color hardly distinguishable from that given when formaldehyde is present.

The fact that the color given by formaldehyde develops much more quickly than that given by proteids alone may also be taken as a rough guide, but is of little value when the acid is in great excess.

SULPHUROUS ACID.

A great deal of attention has been given during the last year by the Division of Foods of the Bureau of Chemistry and the branch laboratories in San Francisco and New Orleans to the determination of sulphurous acid in dried fruit and molasses. In connection with the work, various methods for the determination of sulphurous acid were carefully studied in all three laboratories. In the New Orleans laboratory the work was done by A. Hugh Bryan with the assistance of C. O. Dodge and C. M. Boyles; in the San Francisco laboratory the work was done by Ralph A. Gould, E. J. Lea, and others; in the Bureau of Chemistry at Washington by M. C. Albrech, F. W. Liepsner, P. B. Dunbar, and C. P. Wilson.

It was found that the most satisfactory results were obtained with a long-necked, round-bottomed Kjeldahl flask of 500 cc capacity. Twenty grams was the maximum amount of dried fruit which could be used advantageously. On the addition of water the fruit swelled up so that a larger amount caused difficulty in boiling. In the case of molasses, 50 grams was the maximum amount that could be advantageously distilled. The use of larger samples in correspondingly larger flasks was found to be unsatisfactory.

Foaming, which occurs in both classes of products, appears to be best controlled with paraffin. Sodium chlorid deters the foaming with dried fruit to a certain extent, but the benefit derived from its use does not appear to be uniform. A current of carbon dioxid when introduced above the liquid materially decreases foaming, but when it enters at the bottom of the flask foaming seems to be increased.

It has been suggested by C. S. Ash that the volumetric method be modified by distilling into a normal solution of potassium hydroxid instead of into a standard iodine solution as^a is customary. The experiments by Messrs. Gould and Lea do not indicate that this method is advantageous. The average of the results obtained by it are very slightly higher than those obtained by distilling into iodine, but such results are somewhat irregular and duplicates are not altogether satisfactory. If it is impossible to titrate immediately after distillation the potassium hydroxid method has an advantage, as the oxidation of the sulphite occurs very slowly. Some delay may, therefore, follow the distillation in such case. It is essential, however, that titration follow immediately the acidification of the distillate. In case of distilling into iodine, the distillate should be titrated immediately.

Objections to the volumetric method have been repeatedly made, especially by Horne,^a because of the hydrogen sulphid said to be eliminated from molasses

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 105, p. 125.

on boiling with acid. It has been recommended that the hydrogen sulphid be eliminated by passing the distillate through a trap containing cadmium chlorid or copper sulphate. The results obtained by the use of the trap with both dried fruit and molasses were found to be unsatisfactory. The efficiency of the apparatus was often somewhat reduced, although such reduction did not appear to be due to the presence of hydrogen sulphid, as no deposit of sulphid was found. A slight black deposit was sometimes found in the case of the molasses samples, but examination showed that it did not consist of copper sulphid. It therefore appears to be definitely proven that the amount of the hydrogen sulphid eliminated in the determination of sulphurous acid in dried fruits and molasses is so slight as not to interfere with the accuracy of the operation.

Zerban (page 77) has found that satisfactory results in the determination of sulphur dioxide in molasses can only be obtained by diluting 50 or 100 grams of the sample to 1,000 cc and distilling 800 cc. Even then an additional amount of distillate would contain some sulphur. Bryan and his associates found it unnecessary to employ such dilution or to distil so great a volume. His method was to dilute 50 grams of molasses with 100 cc of water, distill until the foam turns darker, add by means of a separatory funnel 3 portions of water of 50 cc each, and continue the distillation as long as possible after each addition. It was found that practically complete results were obtained by this method.

In the case of dried fruit, Gould found that satisfactory results could be obtained by treating 16 grams of the sample with 175 cc of water and distilling to a small volume. From the consequent concentration more complete results are obtained than from the distillation of a much larger but less concentrated volume.

Practically complete results were obtained in Washington by treating 20 grams of dried fruit with 300 cc of water and distilling 150 cc. Almost all of the sulphur dioxide was contained in the first 100 cc of the distillate, and the amount contained in portions of the distillate following the first 150 cc contained sulphur dioxide in negligible quantity, if at all.

It was demonstrated in all three laboratories that the greatest care must be exercised to prevent charring, especially if the volumetric method is employed. Charring evidently produces a reducing substance which gives a false indication regarding the amount of sulphur dioxide present. Bryan suggests guarding against this by placing the flask upon an asbestos board and exposing but a small surface to the flame. In all three laboratories, and especially in San Francisco and New Orleans, a careful and exhaustive comparison was made of the reliability, efficiency, and accuracy of the volumetric with the gravimetric method. The results obtained with the gravimetric method are slightly higher in the presence of a small amount of sulphur dioxide. In the presence of larger amounts the results are comparative in both molasses and dried fruits. Comparative results with the two methods were obtained in New Orleans with molasses containing 380 milligrams and more of sulphur dioxide per kilogram.

Gould and Lea studied carefully the sources of inaccuracy in the volumetric method which depends on receiving the distillate in iodine and titrating the residual iodine. They found that the chief source of inaccuracy lay in the volatilization of the iodine which is considerable when a current of carbon dioxide is employed. This volatilization was most pronounced when a beaker was used as a receiver, but was excessive even when a flask was employed. Traps were ineffective when attached to an ordinary flask, as cork stoppers were found to absorb quite an amount of iodine and rubber stoppers were still more objectionable.

The best results by the volumetric method were obtained with 8-ounce Drechsel wash bottles, attached to a trap provided with 5 cc of N/50 thiosulphate

solution. By this means the loss was reduced to 0.2 cc of N/50' Iodin solution. The efficiency of the gravimetric method with both peaches and molasses is much greater in the presence of a large amount of sulphur dioxide than in the presence of a small amount. According to the results obtained in Washington, in the presence of 200 milligrams of sulphur dioxide per kilogram of fruit, the efficiency amounts to but 45 per cent; with 500 milligrams, to 70 per cent; with 1,000 milligrams, to from 70 to 75 per cent, and with 2,000 milligrams to about 85 per cent of the sulphur dioxide present. This was as large a percentage as could be recovered regularly with dried fruit. In the New Orleans laboratory the efficiency ranged from 83 to 89 per cent in molasses whose content of sulphur dioxide varied from 100 to 3,300 milligrams per kilogram. As with the dried fruit, the greater efficiency was secured in the presence of the larger amount of sulphur dioxide.

According to the results obtained both at San Francisco and New Orleans, the use of carbon dioxide may be greatly reduced. It can be advantageously employed to remove the air from the flask before beginning the distillation, but the current may then be stopped without sacrifice. Mr. Bryan found that equally good results were obtained by dispensing with the current of carbon dioxide and using instead a solution of sodium bicarbonate, the acidity being increased correspondingly. The purity of the reagent should be tested by making blank determinations for sulphur trioxide, as some blanks will run as high as 50 to 60 mg per kilogram. In the New Orleans laboratory blanks were made daily and the results subtracted from the determinations for that particular day.

LEGISLATION ON PRESERVATIVES IN FOOD.

By H. L. HARRIS, *Pacific Coast Borax Company.*

When William H. Moody was Attorney-General he said: "A thing may be within the statute but not within the letter, or within the letter and not within the statute. The intent of the lawmaker is the law." The pure food law was enacted for the purpose of protecting consumers from being defrauded or injured by adulterated food products, and to compel merchants to truthfully label their goods. Among the matters it aims to regulate is the use of preservatives in food. By authority of this law Food Inspection Decision 76 says: "It has been determined that no drug, chemical, or harmful or deleterious dye or preservative may be used. Common salt, sugar, wood smoke, potable distilled liquors, vinegar, and condiments may be used." It is evident that the food inspectors consider salt, sugar, smoke, alcohol, vinegar, etc., innocent, wholesome preservatives. I do not suppose that they would have us believe that these substances may not, under some conditions, be injurious. In regard to this latter point various authorities express themselves as follows:

SALT.

The United States Dispensary, page 1249: "Sodium chlorid in small doses acts as a stomachic tonic and anthelmintic, in larger ones as a purgative and emetic."

Food and Dietetics, Hutchison, page 291: "It has been asserted, for instance, that the addition of salt to the food aids digestion (Ogata), but more recent and exact experiments have shown that in health, at least, and in moderate doses salt has very little real influence on digestion at all, while in large quantities it actually delays the process."

Practical Hygiene, Harrington, page 209: "In the process of salting, the soluble organic constituents of meat and fish are removed in large part, and the fibers become hardened. The nutritive value and digestibility therefore are diminished correspondingly. Brine salting of fish is one of the oldest processes of preservation known."

Farmers' Bulletin 183, page 29: "Salt is an astringent, and when applied alone to meat renders it very hard and dry. Its action is first to draw out the meat juices. In a few days it will contract and harden the muscle fibers, thus shrinking the volume of meat."

Salt is a natural constituent of the blood. In small quantities it is, without question, beneficial and even necessary to health. When it is used, however, in large quantities to cure or preserve meats, fish, etc., it undoubtedly lessens the flavor, diminishes the food value, and renders food thus preserved or cured more difficult to digest. Unless its use is properly controlled it may be distinctly injurious to health.

SUGAR.

Food and Dietetics, Hutchison, page 275: "In strong solution sugar is an irritant to the tissues. In contact with the skin it is apt to set up superficial inflammation. This is familiar in the form of eczema, which is apt to appear in diabetics from the contact of the sugar-containing urine with the skin, and from the similar condition occurring on the arms of grocers and other persons who have frequently to handle sugar, and it is on account of its irritating properties that sugar can not be used as a subcutaneous aliment. * * * The same is true of the stomach. Brandl, experimenting on dogs, found that a 5.7 per cent solution of sugar produced reddening of the mucous membrane; if the concentration was increased to 10 per cent the mucous membrane became dark red, while a 20 per cent solution produced pain and great distress. This irritating effect on the mucous membrane is accompanied by the production of much mucus and the pouring out of a highly acid gastric juice."

The above evidence clearly indicates that sugar may be injurious to the cuticle or mucous membrane.

SMOKE.

Practical Hygiene, Harrington, page 209: "Smoking consists in exposing the meat or fish to the action of the smoke of wood fires, after, as a rule, a preliminary salting. The exposed material, already deprived of part of its natural moisture, becomes dried still further, and is partly penetrated by acetic acid, creosote, and other preservative elements of smoke."

Preservatives in Food and Food Examination, Thresh and Porter, page 9: "Several articles of food are frequently preserved by being exposed to the smoke from smoldering wood or sawdust. During this process they are partially dried and the material absorbs certain antiseptic substances from the smoke. * * * Creosote is probably one of the active antiseptic agents. It is a very poisonous substance, and doubtless a great outcry would be raised were anyone to attempt to use it for preserving food, but so long as it is introduced into the food in an old-fashioned manner, no objections are raised. It is only when some one wishes to improve upon ancient methods that the effect of prejudice and conservatism makes itself felt. It has never been alleged, so far as we are aware, that smoked meat is unwholesome, though its digestibility is almost certainly impaired. Any modern system of preserving which affected the digestibility to a similar degree would be strongly condemned."

ALCOHOL.

Food and Dietetics, Hutchison, page 343: "The bad effects of alcohol taken in quantities sufficient to produce intoxication are too apparent to require to be insisted upon. It must be remembered, however, that the habitual consumption of alcohol in quantities which, though insufficient to produce any outward and visible signs of intoxication, are yet beyond the immediate oxidizing power of the cells, may end by playing havoc with the tissues. Here, again, the brain seems specially liable to suffer, probably owing to its being one of the most highly organized and delicate tissues of the body. * * * Throughout the body generally the presence of even a slight amount of undecomposed alcohol leads to a diminution of the chemical energy of the cells, which interferes with the ordinary course of metabolism, and may result in chronic disease."

VINEGAR.

Food and Dietetics, Hutchison, page 284: "Although the acetic acid which vinegar contains is ultimately oxidized in the body, with the production of alkaline compounds, there is still reason to believe that it has an unfavorable influence in gout, and may even precipitate an attack if freely indulged in."

Preservatives in Food and Food Examination, Thresh and Porter, page 93: "Vinegar is a very popular condiment and preservative. Taken in excessive quantities it interferes with the digestive processes, and its use in such quantities, if persisted in, ultimately causes emaciation. Used in moderation, however, it is not likely to produce injurious consequences; nevertheless, if it were not one of the oldest preservatives in use, objections would be raised to its introduction. The effect of excessive quantities on adults and children and its potential effect on invalids would be dilated upon, and its use probably condemned."

It follows from the above facts that the preservatives deemed innocent are, under certain conditions, capable of producing distinct injury to the digestive system. The use of certain other preservatives is not permissible under Food-Inspection Decision 76. Among these the boron preservatives are included. In the effort to eliminate harmful substances from food much has been said about the use of boron preservatives, and the conclusion has been reached by some that they are actually harmful. After a most careful, and, as far as possible, unprejudiced consideration of the facts in the case, and after advice with many professionally competent to form an opinion upon this question, it is my honest conviction that boron preservatives are less injurious to health than the substances permitted by Food-Inspection Decision 76. If this is true, the fact will in time be recognized. When it is recognized we will want to make use of the available means that are most efficient. The boron preservatives far outweigh, in their preserving properties, the substances permitted by Decision 76. True, if the question of quantity is ignored, and if conditions not existing in preserved foods are considered, facts may be arrayed against boron preservatives precisely as we have found them in the case of common salt, sugar, wood smoke, potable distilled liquors, and vinegar. But if such facts are not prohibitive against these preservatives of the ancients, why should they be given greater weight against our modern preservatives? While it is true reports have been circulated that boric acid and borax have been injurious to health, when such reports are scientifically investigated the facts invariably show that it was not food preserved with boron compounds, but that excessive doses had been administered medicinally. Boron compounds should not be condemned as food

preservatives on account of excessive quantities having been administered medicinally to invalids.

Let us look critically and honestly at the latest report of an assumed poisonous action of borax. In the *Lancet* of August 10, 1907, page 369, we read: "A fortnight after birth the child developed thrush, for which borax and honey were applied. The child seemed to be relieved of the thrush by this remedy and developed such a liking for it that it was applied most liberally, from two to three four-drachm boxes having been used every week from the second to the eighth week."

The article states that the child wasted away, and that there was no cause for the wasting and rash except the borax, of which the child had about 10 grains every day for six weeks. I do not wish to seem to advocate the administration of borax in this amount to an infant of two to eight weeks, but even with its use does the evidence establish the fact that it acted injuriously? The existing condition of thrush shows that the child was ill before the administration of borax. What was responsible for that? Was the child suffering from a catarrhal stomatitis, perhaps as a result of improper feeding, or was the thrush due to want of cleanliness of the mouth? In the latter event, is it not quite as just to maintain that the child wasted away because of a condition that might have been prevented by the proper use of a solution of borax for keeping the mouth clean? If so, it was the lack of borax before the disease, and not its use afterwards, that was the cause, so far as the borax is concerned; at least, there are two points of view in the matter. If injury was caused by the medicinal application containing borax, may not the honey have been at fault rather than the borax? In speaking of honey, Harrington, in *Practical Hygiene*, page 166, says: "Honey from the flowers of the yellow jasmine has been known to produce serious and even fatal results."

Diet in Health and Disease, Friedenwald and Ruhrah, page 188: "Honey may contain poison. Plugge found that the honey from the *Rhododendron ponticum* is poisonous, and Xenophon, in his *Anabasis*, describes attacks of intoxication due to eating honey. Although death seemed near, none of his soldiers were killed by it. Strabbo and Dioscorides both speak of honey as producing madness or melancholia. * * * The honey from gelsemium is also poisonous. In Branchville, S. C., twenty persons were made ill and three died from eating honey derived from this source. In New Zealand honey from the 'whauriki,' a cress-like plant, causes severe symptoms and sometimes death."

When the poisonous effects of honey are so well known, was it wise to feed a two-weeks-old infant comparatively all the borax and honey it could swallow, and was it right to conclude that borax was the agent that caused the illness? I believe that there is no more authentic evidence than this of boron-preserved foods ever having caused disease in persons because of the boron present. On the contrary, there is ample evidence that there is a vital necessity for the proper preservation of perishable articles of food.

According to the press reports there have been, during the past nine months, over 4,700 cases of ptomaine poisoning in the United States, 119 of which were fatal. There have doubtless been many more that were not reported by the press. All such cases were preventable by proper hygienic measures. Under the above circumstances, is it just to condemn modern methods of preserving food upon what, at most, is uncertain evidence?

I sincerely trust, gentlemen, you will carefully consider the above facts, and realize that food which poisoned thousands of our fellow beings is not pure food, and that it deteriorated and became impure and poisonous owing to the fact

that it was not preserved; and, further, that a judicious use of boron compounds will protect the lives and health of the public by keeping food in a sweet, healthful, hygienic condition. The above facts should appeal to you so forcibly that you will at once realize the value, necessity, and innocence of boron compounds when used for the preservation of food.

The following committees on recommendations of referees were appointed by the president:

Committee A.—R. J. Davidson; Harry Snyder; H. D. Haskins.

Committee B.—B. B. Ross; F. D. Fuller; Harry Snyder.

Committee C.—L. M. Tolman; A. L. Winton; A. McGill.

The convention adjourned to meet at 9.30 Thursday morning.

SECOND DAY.

THURSDAY—MORNING SESSION.

At the opening of the session the following papers were presented, which, together with the report of the associate referee on the determination of water in foods, completed the subject of food adulteration.

Mr. C. B. Cochran submitted an outline of the classification of coal-tar colors to serve as a basis for future investigations, with the idea that, if possible, a committee be appointed to assist in the prosecution of such an investigation. In this connection the following paper by Mr. Cochran is also submitted:

THE ACTION OF SODIUM BISULPHITE REAGENT ON CERTAIN COAL-TAR COLORS.

By C. B. COCHRAN.

The sodium bisulphite reagent is made by saturating a 5 per cent solution of sodium hydroxid with sulphur dioxide. It is therefore a solution of $\text{NaHSO}_3 + \text{H}_2\text{SO}_3$. This reagent added to a water solution of any of the following coal-tar colors, decolorizes the solution, and on heating the color reappears but disappears again on cooling. The heating and cooling may be repeated many times and each time the color will reappear in the solution while hot.

Acid magenta (A) S. and J. 462.

Fuchsin (A) S. and J. 448.

Methyl violet (Grübler) S. and J. 451.

Ethyl violet (Grübler) S. and J. 453.

Guinea violet 4B (A).

Ethyl green (A) S. and J. 428.

Guinea green B (A).

Chrome green S. and J. 443.

Acid green (Grübler) S. and J. 435.

Malachite green (Grübler).

Turquoise blue BB.

Acridin red (Grübler).

The references to Schultz and Julius are taken from the 1904 edition. (A) indicates the Berlin Aniline Company.

With the exception of acridin red, all the colors in this list belong to Group X of Schultz and Julius which comprises the triphenyl methane and diphenyl-naphthyl methane coloring matters. The list is taken from about two hundred colors examined and includes all those which were found to exhibit the peculiar behavior with sodium bisulphite reagent which has been briefly described.

The only color of Group X thus far examined which has shown a different behavior with sodium bisulphite reagent is China blue (Grübler) S. and J. 480. This color is readily bleached, but the color does not seem to return on heating.

The results of the experiments made with these colors proved that the temperature at which the decolorized solution began to show a return of the color depended on the amount of sodium bisulphite reagent used. It was also found that, in many cases at least, if this reagent was added in considerable excess of the amount required to bleach the solution, no color would return even on heating to the boiling point.

The following description of the results of some of the experiments made on two of the colors will illustrate in a general way the behavior of the above-named coal-tar colors when treated with this reagent:

Acid magenta (A).—A solution of this color was made of such a depth that printed letters one-eighth of an inch long could just be distinguished in strong daylight through a layer of 5 inches.

Experiment I: To 5 cc of this solution was added 1 cc of sodium bisulphite reagent. The solution was decolorized within a few seconds. On heating to the boiling point only a faint color returned, which quickly disappeared on cooling.

Experiment II: To 5 cc of solution 0.5 cc of the reagent was added. The color disappeared in two or three minutes. On heating to the boiling point a deep color returned, which disappeared at 50° C.

Acridin red (Grübler).—The solution used was made of the same depth as in the case of acid magenta.

Experiment I: To 5 cc of solution 1 cc of reagent was added. The color disappeared at once. Only a faint color returned on heating to the boiling point.

Experiment II: To 10 cc of the solution 0.1 cc reagent was added. The color disappeared in one minute, but returned in its original intensity on heating to the boiling point and did not entirely disappear until the solution was cooled to 27° C.

Ethyl green (A).—This color is very easily bleached, and if a great excess of the reagent is used no color returns even on heating to the boiling point and continuing the heat for some time. When only a slight excess of the reagent is present a strong green color returns on heating to 60° C., which gradually disappears as the solution cools.

The temperature at which the color finally disappears from heated solutions of any of the colors given in the list depends on the rate of cooling. If cooled slowly the color will disappear at a much higher temperature than if cooled rapidly.

With the majority of the colors given in this list, a solution of sulphur dioxide in water will produce results similar to those obtained with the sodium bisulphite reagent. However, in working with a large number of coal-tar colors the latter reagent was found to give distinctive reactions in a larger number of cases than the sulphur dioxide solution. Preference was therefore given to the bisulphite solution as a reagent for general use.

THE DETERMINATION OF MOISTURE IN SIRUPS AND MOLASSES.

By W. D. HORNE.

The correct determination of water in sirups and molasses is very important both from the technical and the legal point of view—first, because this alone enables one to determine the true purity of the solution, and, secondly, because a sirup is defined by law as a solution containing not more than 25 per cent of water.

Various methods have been proposed for determining the water, but difficulties have been met with in all of them. Undiluted sirups dry slowly and frequently incompletely, necessitating prolonged heating or high temperatures, both of which tend to decompose organic matter. To dilute and mix with sand,

pumice, etc., solves this difficulty only partly. Vacuum drying is not always convenient. It necessitates elaborate apparatus and is often rather slow. The importance of finding a simple, quick, and accurate method for making this determination is apparent, and a very satisfactory procedure, gradually evolved by the writer in the course of many years of experimenting, is offered for the consideration of the association. The method reads as follows:

One gram of molasses is weighed into a flat-bottomed dish 3 inches wide and about 0.5 inch deep and containing a glass rod. About 0.8 cc of water is well mixed with the molasses and then about 30 grams of dry quartz sand, previously extracted with hydrochloric acid, is exactly weighed and added to the diluted molasses. The dish is placed on an open boiling water bath and stirred carefully and frequently during half an hour. This causes the free evaporation of about nine-tenths of the water at a moderate temperature. The dish is next placed in a water-jacketed air bath, where it is heated at the temperature of boiling water for two hours. After cooling it is weighed and reheated for one-hour intervals until the weight is constant.

This very easy method, requiring only the simplest devices and manipulations, gives, in from two to four hours, constant results that agree closely with determinations effected in partial vacuo during longer heating periods and under the influence of a fine stream of air dried by sulphuric acid. The method has been employed on refinery molasses for several years with great satisfaction.

In this work it is found that the degree Brix of the undiluted molasses plus the moisture determined as above amounts to 103.3 with great regularity, despite rather wide variations in the percentage of ash in the sirups.

Mr. Tolman submitted by title a report entitled "A study of the changes taking place in whisky stored in wood," by C. A. Crampton and himself. This report covers an investigation of eight years' duration conducted in the laboratory of the Bureau of Internal Revenue and is published in full in the Journal of the American Chemical Society for January, 1908, page 98. The conclusions reached are as follows:

(1) There are important relationships among the acids, esters, color, and solids in a properly aged whisky, which will differentiate it from artificial mixtures and from young spirit.

(2) All the constituents are undergoing changes as the aging process proceeds, and it is evident that the matured whisky is the result of these combined changes.

(3) The amount of higher alcohols increases in the matured whisky only in proportion to the concentration.

(4) Acids and esters reach an equilibrium, which is maintained after about three or four years.

(5) The characteristic aroma of American whisky is derived almost entirely from the charred package in which it is aged.

(6) The rye whiskies show a higher content of solids, acids, esters, etc., than do the Bourbon whiskies, but this is explained by the fact that heated warehouses are almost universally used for the maturing of rye whiskies, and unheated warehouses for the maturing of Bourbon whiskies.

(7) The improvement in flavor of whiskies stored in charred packages after the fourth year is due largely to concentration.

(8) The oily appearance of a matured whisky is due to material extracted from the charred package, as this appearance is almost lacking in whiskies aged in uncharred wood.

(9) The "body" of a whisky, so called, is due largely to the solids extracted from the wood.

DETECTION OF THICKENERS IN ICE CREAM.

By G. E. PATRICK.

The thickeners commonly used for ice cream to-day are gelatin, certain vegetable gums or jellies, and various forms of starch. Of the true vegetable substances gum tragacanth is most used, and it is believed that at least one other substance of this class is employed, but it was impossible to tell whether it was agar or some less common member of the group.

A test for the detection of such thickeners has been formulated which it is believed will prove useful if the conditions are closely observed, the main features of the method offering little that is new from a chemical point of view. Picric acid is used for precipitating gelatin, and alcohol for the gums. Preparatory to the test the liquid is clarified by coagulating the proteids with acid and heat and filtering. The details of the procedure, elaborated with the help of H. S. Bailey and B. McClelland, of the Dairy Laboratory, are as follows:

To 50 cc of ice cream add 25 cc of water, boil for half a minute to dissolve any thickener that may be present, add 2 cc of a 10 per cent solution of acetic acid, heat again just to boiling, add two or three heaping teaspoonfuls of kieselsguhr, shake well, and filter immediately through a plaited filter. To 3 cc of the clear filtrate add 12 cc of 95 per cent alcohol and mix; this precipitates the milk proteids not removed in the clarification, together with the gums, and some of the gelatin, if much be present; add 3 cc of acidified alcohol prepared by mixing 95 cc of 95 per cent alcohol and 5 cc of concentrated hydrochloric acid; this dissolves the milk proteids completely. If the liquid is clear after this treatment, no gums or vegetable jellies are present. But, on the other hand, turbidity or a precipitate does not necessarily indicate the presence of a thickener; for this may be caused by the large amount of gelatin present or by eggs (more than three or four per gallon), or because the ice cream has become sour. Fortunately the precipitate due to gelatin or to the substance derived from eggs can be readily dissolved by a moderate dilution of the alcohol with water, for instance, by 3 cc of water in the test just described. This amount of water does not sensibly dissolve any precipitate due to vegetable gums or jellies, but entirely dissolves that due to gelatin and eggs. If gum tragacanth is present, the precipitate will be cohesive and stringy, or will become so upon shaking; while if the undissolved matter is due to other vegetable thickeners (possibly agar), it is finely flocculent and devoid of cohesive property.

If the ice cream is sour, there is sometimes a precipitate of another character, which must be derived chiefly, if not entirely, from the cane sugar present, since it appears very faintly, if at all, in tests upon unsweetened milks that have been allowed to sour. But this substance does not always develop in sour ice creams or in sweetened milks that have become sour; its appearance seems to depend upon some special property or condition of the milk, probably upon the presence of certain kinds of bacteria. Its nature is now being investigated, and Mr. C. A. Browne suggests that it may prove to be dextran. Whatever it may be, the precipitate which it yields with alcohol is not sensibly dissolved either by the acidified alcohol or by the water added to dissolve the gelatin and egg substance; therefore it must remain mixed with the vegetable gums and jellies. It does not resemble gum tragacanth, as it is not stringy, but does closely resemble the other vegetable thickener. It is therefore a serious obstacle in the test. Fortunately, however, its formation appears to be prevented by formaldehyde (experiments on this point are still in progress), and it is believed that if fresh ice cream is treated with a liberal dose of formalin there will be no trouble from this annoying substance even if the test is carried out several weeks later.

The test for the detection of gelatin was made on a small portion of diluted cream after boiling but before acidifying to precipitate proteids. The provi-

sional method of the association, originally published by Stokes (Analyst, 1837, p. 320), was used, which consists in clarifying the milk or cream with dilute mercuric nitrate solution and precipitating the gelatin in the filtrate with picric acid. It is in general a satisfactory test. But the interesting fact was observed by Mr. Bailey that in testing ice cream which had been sour for a week or more, or very sour milk, whether previously sugared or not, picric acid produces a yellow precipitate easily mistaken for that produced with gelatin. No way of distinguishing between the two has been found, but, as would be expected, formaldehyde prevents the formation of this "pseudo gelatin" and therefore samples thoroughly preserved with formalin present no difficulty from this source.

Starch, often used in a mixture with gum tragacanth, and sometimes alone, was detected in the usual way with iodine, using a portion of the boiled diluted sample.

REPORT ON THE DETERMINATION OF WATER IN FOODS.

By F. C. WEBER, *Associate Referee*.

The referee regrets that a full and definite report on this subject can not be given this year owing to lack of cooperation and of sufficient time to devote to the work. Circular letters were sent to 15 chemists asking for their collaboration; only 5 replied, stating that they could not take up the work this year.

The results obtained this year showed that drying in a vacuum of 0 to 5 mm of mercury over sulphuric acid compared favorably with drying in an oven with partial vacuum at 100° C. The results, however, in the vacuum oven were obtained in ten hours, while nine days were required to get the same results in the vacuum desiccator. The addition of phosphoric anhydride as an auxiliary drying agent in the vacuum-desiccator method did not appear to shorten the time of drying.

From the somewhat limited amount of work which has been done so far, the referee does not feel justified in condemning the method, though it is doubtful whether it could, even when modified, come into general use. The importance of further study on this subject is recognized, and it is therefore respectfully recommended that the work be continued next year.

THE CARBON DIOXID VALUE OF PURE COMPRESSED YEAST AND COMPRESSED YEAST AND STARCH COMPOUNDS.

By T. J. BRYAN.

The examination of numerous samples of compressed yeast in this laboratory showed that the majority of them contained added starch, usually either potato or corn starch.

The question arose, What is the value of the pure compressed yeast to the consumer as compared to the article mixed with starch? Some preliminary experiments were made, using samples of pure compressed yeast and yeast containing corn starch, such as were found on the market. In these preliminary tests the different yeasts were allowed to act upon a 10 per cent sugar solution and the volume of gas generated was measured. The results showed that the pure yeast had a greater carbon dioxide value than yeast mixed with starch. Owing to the size of the apparatus necessary for collecting and measuring the volume of the gas produced, it was thought better, in subsequent experiments, to determine the amount of carbon dioxide gas produced by the loss in weight. Furthermore, as the different yeasts tested were made by different manufacturers from different cultures, it was decided that the results secured were of little value in deciding the effect of the presence of starch in compressed yeast, and that samples of yeast from the same culture must be

secured, a portion of which should be kept in the pure state, and other portions mixed with potato and corn starch.

Seventy pounds of this pure yeast were mixed with 10 pounds of corn starch and 16 pounds of water (it was found necessary to add the water to make the mixing uniform and in order to produce a cake). To another portion of 70 pounds of the yeast were added 10 pounds of potato starch and 19 pounds of water. To the remaining portion of pure yeast no starch was added in the mixing. These three samples were then pressed separately and cut into 1-pound cakes which were each dipped in water before being wrapped, as is the custom of the manufacturer. These three samples were then allowed to act upon 100 cc of a 10 per cent sugar solution to which 75 cc of water had been added in an Erlenmeyer flask of about 190 cc capacity. This flask was provided with a calcium chlorid tube adjusted by means of a cork in order to prevent loss of weight by evaporation. Two grams of yeast were used in each test and duplicates were run. The flasks were put in a box, the opening of which was covered with a towel and placed against the chimney where a temperature varying between 20° and 30° C. was maintained. During the last three days the box was replaced by a section of a bookcase and the temperature maintained ran a little higher than on the preceding days, varying between 25° and 30° C. Owing to the varying temperatures on different days only the results for the same periods on the same day are comparable. The flasks were weighed when they had been filled with the reagents and once every hour after that for ten hours, and again at the end of each twenty-four hours. Acknowledgments are due to Messrs. Nehls and Gardner and Mrs. M. B. Shulda for assistance in making the tests.

Table I shows the average loss in weight for the periods specified. Table II shows the average per cent of loss in weight (or the percentage of the weight of carbon dioxid gas produced for the specified periods), considering that the average weight of carbon dioxid produced by the pure yeast samples for each period is 100 per cent. It will be noted that after two hours the average loss in weight of the flasks containing pure yeast is, with a single exception, greater at every weighing.

The average percentages of the yield of carbon dioxid gas for twenty-four hours and for six hours, as given in Table II, show conclusively that the effect of starch upon yeast is to reduce the carbon dioxid value and that this percentage reduction is greater than the percentage of starch used in preparing the samples.

It has been claimed by some manufacturers that it is necessary to use starch in compressed yeast in order to preserve the same. The data in Table II show that on the fourteenth day the value of the pure yeast is greater than on the first day as compared with both of the starch yeast mixtures. The differences, however, are not sufficient, in the writer's opinion to justify the statement that the yeasts containing starch had deteriorated, though the data point in that direction. As fourteen days is a longer time than compressed yeast is kept before being put on the market and used the contention that starch is necessary to preserve yeast is seen to be absolutely false. Nineteen days after the yeast was prepared all of the samples were perfectly sweet.

It was thought desirable to test the action of these different yeasts in the making of bread. The bread was made with 650 grams of flour, 500 grams of water, and 10 grams of yeast or yeast starch mixture. On the second day pure yeast yielded bread having a volume of 2,225 cc; corn-starch yeast mixture, bread 2,100 cc in volume. On the fifth day pure yeast yielded bread of a volume of 2,000 cc; corn-starch yeast yielded bread 1,860 cc in volume. On the twelfth day pure yeast yielded bread 2,150 cc; corn-starch yeast mixture, bread 2,015 cc. On the thirteenth day pure yeast yielded bread 3,000 cc in volume and corn-starch yeast mixture, bread of 2,575 cc. On the fourteenth day the

bread from the pure yeast had a volume of 2,820 cc, while the corn-starch yeast mixture bread had a volume of 2,650 cc.

As in the action of yeast on sugar solutions, the actual baking tests show that the carbon dioxide value is always greater in the case of pure yeast. By comparison it will be found, however, that the average percentage difference in the size of the loaves is less than the average difference in loss of weight of the sugar solutions for the same periods. This is due to the fact that in the case of the bread it is only the final volumes that are compared, whereas to make the results comparable with those obtained by the action of yeast on sugar solutions it would be necessary to determine the difference in volume (for 2 grams of yeast) between the freshly mixed bread and the final volume of the loaf.

There is much work to be done before the action of starch in yeast will be known in all its details. The writer feels, however, that the results here given are sufficient to justify the statement that starch in compressed yeast is an adulteration. The advantage to the manufacturer is easily seen when we consider that he sells starch which costs less than 3 cents per pound at the price of yeast which costs 15 cents per pound. A standard for compressed yeast that will exclude the use of added starch is therefore most desirable. Next to the quality of the flour the quality of the yeast is of prime importance to the 70,000,000 people of this country in the preparation of their bread.

TABLE I.—Average loss in weight of sugar solution containing pure yeast and yeast-starch mixtures, September 19 to October 2, 1907.

Number of hours.	1 day old.			2 days old.			5 days old.		
	Pure yeast.	Potato starch mixture.	Corn-starch mixture.	Pure yeast.	Potato starch mixture.	Corn-starch mixture.	Pure yeast.	Potato starch mixture.	Corn-starch mixture.
	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>
1	0.035	0.012	0.028	0.039	0.013	0.019	0.027	0.034	0.033
2	.081	.045	.063	.071	.042	.055	.089	.082	.091
3	.185	.127	.170	.193	.129	.150	.300	.240	.239
4	.325	.232	.288	.314	.216	.239	.404	.318	.375
5	.548	.413	.437	.524	.372	.360	.587	.506	.572
6	.770	.610	.631	.664	.495	.519	.824	.665	.754
7	.888	.719	.757	.849	.678	.687	.916	.753	.873
8	1.064	.874	.920	1.008	.819	.795	1.072	.884	.985
9	1.222	1.022	1.075	1.136	.914	.903	1.181	.972	1.100
10	1.329	1.115	1.170	1.206	1.003	1.011	2.520	2.230	2.378
24	2.779	2.345	2.415	2.661	2.189	2.313			
	6 days old.			7 days old.			8 days old.		
1	.047	.051	.031	.018	.023	.037	.051	.047	.044
3	.163	.148	.126	.158	.120	.146	.158	.130	.127
4	.373	.315	.280	.268	.200	.217	.325	.233	.252
5	.601	.517	.452	.533	.409	.461	.525	.410	.409
6	.742	.616	.613	.748	.570	.645	.695	.522	.542
7	.982	.817	.797	.930	.710	.789	.850	.682	.683
8	1.068	.915	.907	1.082	.829	.915	1.025	.798	.826
9	1.228	1.026	1.024	1.202	.937	1.027	1.129	.878	.916
10				1.326	1.037	1.123	1.206	.987	1.025
24	2.645	2.334	2.279	2.662	2.167	2.392	2.517	2.041	2.167
	9 days old.			13 days old.			14 days old.		
1	.034	.041	.029	.039	.039	.031	.022	.016	.019
2	.193	.174	.123	.199	.131	.105	.127	.086	.088
3	.408	.376	.332	.391	.240	.254	.279	.188	.197
4	.642	.538	.492	.631	.442	.458	.513	.375	.398
5	.813	.690	.631	.816	.593	.600	.721	.537	.564
6	.969	.795	.768	1.015	.750	.771	.885	.674	.707
7	1.119	.904	.912				1.093	.847	.883
8	1.322	1.055	1.043				1.218	.950	.988
9	1.469	1.170	1.164						
21				3.120	2.460	2.517			
24	2.970	2.522	2.392				3.134	2.546	2.655

TABLE II.—Carbon dioxide gas produced in sugar solutions containing yeast-starch mixtures as compared with those containing pure yeast, rated at 100.

Age of yeast.	For twenty-four hours.			For six hours.		
	Pure yeast.	Potato starch mixture.	Corn-starch mixture.	Pure yeast.	Potato starch mixture.	Corn-starch mixture.
Days.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
1.....	100	84.4	86.9	100	79.2	81.9
2.....	100	82.3	86.9	100	74.5	78.1
5.....	100	88.5	94.4	100	86.0	97.4
6.....	100	88.2	86.2	100	83.0	82.6
7.....	100	81.4	89.9	100	76.2	86.2
8.....	100	81.1	86.1	100	75.1	77.9
9.....	100	84.9	80.5	100	91.6	88.4
13.....	100	78.8	80.6	100	73.9	76.0
14.....	100	81.2	84.7	100	76.2	79.9
Average.....	100	83.4	86.2	100	79.5	83.1

Vice-President Snyder took the chair at this point and Mr. J. P. Street delivered the annual presidential address.

PRESIDENT'S ADDRESS.

By J. P. STREET.

Gentlemen of the Association: An unfortunate unwritten law, supported by the custom of many years, demands an address from your presiding officer. My 23 predecessors have covered the ground so well, and have appreciated the merits and the failings of this association so thoroughly, that there is but little left for me to do save to repeat their congratulations and emphasize their warnings.

That the original object of this association was to secure uniform and accurate methods for the analysis of fertilizers is known to you all. Likewise you are all familiar with our steady growth, until now we include within the province of our study and control practically all materials connected with agricultural industry. This widening of the association's activities has limited in some degree the interest of the members in its work as a whole. This is an inevitable result of our expansion, and, deplorable as it is from certain points of view, it could not be expected that either time, inclination, or immediate interest would lead all the members to take part in all the work. This is no reason, however, why every member should not take part in some of the cooperative work each year, and by his careful work help in the solution of the numerous problems that still confront the agricultural chemist. In the association's earlier days it was the custom for practically every institution represented in its membership to share in the cooperative work, and this was easily possible when fertilizers alone were the subject of study. It was also the custom then that this work should be performed by the experienced men of the staff, so that when the results were presented to the referee for comparison and study they would furnish a reasonable test of the method's accuracy, and not of the capability of the analysts. But in later years this condition to a great extent changed; the official samples all too often were used to check the younger assistants rather than the method itself. This resulted in certain cases in injustice to methods which proved later to be thoroughly dependable, and it would seem that the publication of certain of the comparative results must have had a harmful effect on our standing as an association. It is a matter of deep gratification,

therefore, to observe that as a result of recent agitation of this subject the pendulum is swinging back, and we find that a large proportion of our association work is again intrusted to those best qualified to handle it. While the privilege of assisting in our work should be denied to none of our members, still greater efficiency would be secured if even wider latitude were permitted the referees in the selection of their associates. These associates should be not only skilled manipulators, but should have given particular attention to the subject under investigation. While general cooperation, as I shall point out later, is most desirable, it would be far better for the skilled chemist to decline to take part in an investigation than to intrust the details to an inexperienced assistant. Surely there is no member of this association who is not expert in at least one branch of agricultural analysis and whose work would not be of great value if properly directed.

As a matter of interest, I have tabulated the work of the past ten years, to determine the amount of cooperation of the various State laboratories. In preparing this summary I have naturally left entirely out of consideration the Bureau of Chemistry, and it is only just that attention should be called to the valuable work done by the Bureau each year in the study of our methods. With five referees and eight associates included in its organization this year, besides doubtless many others assisting in the work of other referees, the interest of its controlling officers in this association is manifest, and we are under deep obligation to them for the liberal policy which they have fathered. When we consider the results of my tabulation, and learn what the attitude of the various State institutions has been toward this association, a different story is told. I find that two of the States took part in the work in each of the ten years, thirteen in from seven to nine years, fourteen in from four to six years, three in from one to three years, and four States gave no assistance whatever. Four States, three of them distinctively agricultural, showed not the slightest practical interest in the work of the association during a period of ten years. By further subdividing the time under consideration into two periods of five years each, it is seen that there has been an increased activity in all the groups of States during the past five years, with the exception of the Middle West, this activity being specially marked in the Central and Pacific States. The encouragement furnished by this second classification is somewhat diminished, however, by the fact that from 1901 to 1906, inclusive, six States cooperated but twice, thirteen but once, and nine not at all. Do not these facts, which can be easily verified by a study of our reports, show either that there is a lack of sympathy with the association and its work on the part of those in control of many of our experiment stations, or that there is a willingness to cast the burden of this work on others, and enjoy the fruits of their labors. Efficient and accurate methods of analysis are the groundwork of all agricultural research, and any station which fails to aid in their development and application shirks a manifest duty and plays the rôle of a parasite on this association.

There is another subject which I feel many of our members do not approach with a keen sense of the duty involved. In 1896, when acting as referee on nitrogen, I made a recommendation, which after much discussion by the committee was distorted to read as follows: *Resolved*, That it is the sense of this association that the forms of the nitrogen in commercial fertilizers should be reported, where it is possible to do so." The clause "where it is possible to do so" cast the resolution, as far as its effect was concerned, into

"A limbo large and broad, since called
The paradise of fools."

In 1902, as chairman of Committee A on Recommendations, I was able to rescue the matter in a measure from its undeserved oblivion by urging the association to take a somewhat more decided stand by the adoption of the following resolution: "That it be earnestly urged that, in reporting the analyses of complete fertilizers, the amounts of nitrogen existing in the form of nitrates, ammonia salts, and organic nitrogen be specifically stated." And there the matter rests to-day.

In spite of this specific recommendation from what should be considered the court of last resort in matters pertaining to agricultural chemistry, what has been the attitude of those having charge of the thirty-one different fertilizer controls? Five controls report the three forms of nitrogen, three report nitrogen soluble and insoluble in water, or total nitrogen and nitrates, and one reports total and available nitrogen; twenty-two report only total nitrogen. Fifteen of these delinquents report phosphoric acid in three forms, and four of them separate the potash, as from chlorid and sulphates. Is not this a glaring inconsistency? The form of the nitrogen, the most important and most expensive ingredient of the fertilizer, is absolutely ignored by 70 per cent of the inspection officials, while insoluble phosphoric acid is carefully determined by most of them by a method whose accuracy is universally discredited. Surely there is no excuse for this careless, I will not say ignorant, attitude. Excellent, exact, and rapid methods for the determination of nitrates and ammonia salts are at the disposal of every inspection official, and the resulting benefits would more than compensate for the slight additional time consumed.

The only excuse that I have ever heard offered for failure to make these determinations was lack of time to do more than the law required. I can not believe this an honest excuse. Is it not time that the fertilizer appropriation or the income from fertilizer fees should cease to be considered as a financial aid to carry on more interesting investigations in other lines of work? Is it not our official duty, not simply to make a perfunctory examination to satisfy the requirements of the law, but also to impart to the consumer all the information possible from our present methods of analysis as to the sources of the materials used in compounding the mixtures offered to him for purchase? And what has been the result of this indifference, this neglect of duty, this shirking of responsibility? The fertilizer manufacturer has watched us, gentlemen; he has learned the weak points in our armor, and he has always kept a decade in advance of us. The commercial instinct is not dormant in his heart. The high price of nitrogenous fertilizing materials has tempted him to use any and all materials, often inert and valueless, for he knew that with few exceptions the inspection officials would not differentiate these from higher grade materials, but would by their analysis classify them with the best and most available forms, and give them a correspondingly high commercial valuation. Is it any wonder, then, and are we entirely blameless, that the use of these inert materials is continually increasing, until now we are forced in defense of the interests confided to our care to attempt some laboratory method for distinguishing available and inert organic nitrogen? For several years the association has been studying methods with this end in view, but the progress has been slow and the cooperative work has been slight. It is a subject which merits the earnest thought and the patient study of every fertilizer control official, and without this concentrated effort the problem will long remain unsolved. Our obligation to the manufacturer who continues to use only high-grade materials in his fertilizers is as well defined as is our duty toward our constituents, the agricultural public, who

have a right to know the nature of the materials offered them as possessing high fertilizing value.

A review of the history of this association clearly indicates that it is a conservative organization. This conservatism has always been an element of strength, and the wisdom of our attitude toward the adoption of new methods can not be questioned. When we have been confronted by new agricultural problems, however, it must be confessed that in some cases at least our conservatism has degenerated into a sort of artful dodging. Two queries well illustrate my contention: How shall we measure the availability of basic slag? And is there such a thing as "available" potash as distinguished from water-soluble potash? Our attitude toward these problems is known by you all. Basic slag has been the shuttlecock of this association for a number of years, and, as far as any official pronouncement as to its status and methods to determine its worth is concerned, we are just about where we started. The importance of this material as a phosphatic fertilizer will increase with the years, and the time is not far distant when we shall be obliged to decide the question by some other method than skillful side stepping. The question of "available" potash is being forced on our attention by the manufacturers, who make the twofold claim that our present methods do not credit them with all the water-soluble potash they supply in their mixtures and do not recognize the agricultural value of certain forms of potash which are insoluble in water. The work of our referee on potash for the past two years seems to support the contention that not all the water-soluble potash added appears in the analysis when made by the official methods. It is true that we may have serious doubts as to the wisdom of adopting any modification of the method which permits the use of a solvent, which might bring inert potash into solution, and I believe the association made no mistake in rejecting the recommendation of the referee for the adoption of this modification. Nevertheless the manufacturer, who surely is entitled to justice and protection quite as much as the consumer, makes a claim which our own work seems to justify, and the obligation rests upon us to seek some modification of our methods which will remove from them even the slightest stigma of injustice. And the same holds true with the question of "available potash." We are not in a position to affirm or deny the manufacturer's claim that the potash which he adds in an organic or mineral form, and which is not soluble in water, possesses an agricultural value approximately the same as the water-soluble form. The claim, however, demands our consideration and is worthy of our careful study.

We must not deceive ourselves into the comforting belief that the labors of this association are at an end. In spite of the remarkable progress made by our very efficient referees in the last few years in perfecting our methods for food analysis, every food chemist realizes the gaps which still exist in these methods and the new problems that are constantly being presented. The analysis of drugs is almost an unexplored field. Your attention has just been called to some of the unsolved problems in fertilizer analysis. A brief glance at the subject of cattle foods will show that here is another unexhausted territory. It is hardly necessary to call attention to the fact that no one of the ordinary determinations in cattle foods, with the possible exception of ash, is scientifically accurate. These inaccuracies are, however, in themselves of minor importance from a feeding standpoint, except that the algebraic sum of all the errors involved, by our present method of calculation by difference, falls upon the "nitrogen-free extract." Not only is the aggregate percentage conceded to "nitrogen-free extract" usually inexact, but it is manifestly unscientific to group together under one heading such dissimilar substances as

sugars, starch, gums, pectin bodies, pentosans, lignin, and the various modifications of the hemicelluloses and oxycelluloses, especially when they constitute such a large part of the feed under examination, varying from 26 per cent in cotton-seed meal to 80 per cent in rice. The meaninglessness of this term has been recognized by all, and in recent years much excellent, and in some cases conclusive, work has been done toward the separation and determination of many of these carbohydrate bodies. Recent work of my own in a study of the carbohydrates of dried beet pulp has convinced me that this matter is important not only from an analytical point of view, but also in that its solution would shed much light on many of the problems of animal feeding. The sugars and starches are by common consent rated as the most assimilable forms of the carbohydrates, and yet feeding experiments have shown that beet pulp, which contains these forms in minimum quantities, was more efficacious than hominy meal, a typical starchy feed. Does not this surprising result indicate either that our methods of feed analysis are misleading, or that certain of the carbohydrates generally classed as inferior for feeding purposes are, in certain feeds at least, of superior quality and of high digestibility?

By subjecting beet pulp successively to a series of solvents, as originally suggested by Dragendorff, and further developed by Parsons, Frear, Browne, Sherman, and others, and analyzing the various residues and solutions, I secured some very interesting and suggestive results. Large amounts of araban, galactan, lignin acids, and lignin were found, with smaller quantities of pectin, parapectin, and reducing and invert sugars; while nearly one-quarter, or 16 per cent, of the carbohydrates existed as unidentified hemicelluloses, which were dissolved by dilute sulphuric acid and were present in an incompletely hydrolysed state. It was shown that the galactan was present in three forms; one fraction was soluble in 80 per cent alcohol, one was soluble in cold water, and the balance was hydrolysed by dilute sulphuric acid. The pentosan arabans in this case were even more complex, and varied greatly in their resistance to solvents. Varying portions were soluble in cold water, in dilute acid, in warm water, in diastase solution, and in dilute alkali, and still another portion was removed by chlorination. The chlorination process gave a percentage of cellulose 6.5 per cent lower than the official method, the residue being entirely free of pentosans and proteids, but still containing one-fourth of the original ash.

Time does not permit me to go into further details as to the results of this investigation. My experience with this material, however, leads me to the conclusion that for a rational basis of scientific feeding we must not be content with the determination of the total of any ingredient, but must differentiate its various forms as indicated by their behavior with a series of solvents. The fact that we have in beet pulp apparently at least six different modifications of araban and three of galactan would encourage the assumption that a determination of total araban and total galactan in a feed conveys as little useful information as one of total phosphoric acid in a fertilizer.

On reading the constitution of this association I note that its objects are two-fold, not only to secure uniform and accurate methods of analysis, as already mentioned, but also "to afford opportunity for the discussion of matters of interest to agricultural chemists." It would seem that this second and very important object of the association had been to a great extent neglected. We have increased the scope of the association without a corresponding increase in the length of our meeting, with the result that practically all of our time has been occupied in the presentation of reports, too often presented in an undigested form, which because the necessary data are not before us, are unin-

telligible to all except the few who have taken part in the work on the particular subject in question. Discussion of the reports has been reduced to a minimum; in fact, it has been discouraged. As a result, our meetings have lost some of their interest, and the benefits resulting from them have somewhat diminished. No member of this association, I am confident, will deny that one of the greatest advantages derived from attendance at these meetings is the opportunity for the exchange of ideas, whereby we learn the experience of others in the lines of work that have presented difficulties to us ourselves. This information we have been forced to obtain outside the formal meetings of the association, and the benefits accruing therefrom have therefore been limited to those whose intimate acquaintance has permitted questions a stranger would not feel at liberty to ask. It would seem that these benefits derived from experience should be open to all our members, and that the proper place for their discussion is the floor of this convention.

It is impossible for us all to take an equal interest in all the ramified work of this association, and a general meeting for papers and discussion might not meet with unqualified success. I would urge, therefore, that one day be devoted entirely to the reading of papers and their discussion, and that for this purpose the convention be divided into three sections covering the same subjects as our present committees on recommendations. All papers to be read in these sectional meetings should be referred to the proper committee with full power to reject the same, or assign places to them in the programme, which should be sent to all members prior to the convention. I believe in this way, gentlemen, that the interest in our meetings would be greatly intensified, and an immediate as well as lasting benefit conferred upon our members.

The constantly changing personnel of this association is an element of weakness and gives to it a character of impermanency. This fact has been particularly impressed upon me during the past two years in my work with the committee on revision of methods. We were confronted by a great mass of methods couched in as many forms of English as there had been referees, with varying nomenclature and with no fixed or systematic method of treatment. Under the conditions attending the origin of our methods no other result could have been expected. Your committee has attempted, and I trust with a considerable degree of success, to bring order out of this chaos and has rewritten the methods with the idea of clearness and uniformity of statement constantly in mind. And yet all this labor will go for naught and our methods in a few years will return to their previous state of indefiniteness unless our present system is corrected.

The one permanent office in this association has been that of secretary. There seems to be an unwritten law that during his natural life Doctor Wiley shall be our secretary, his occupancy of that office being conditioned only by his good behavior. The benefits accruing to the association by this, its one permanent feature, are undoubted, and any effort to insure further permanency in other departments of the association would seem worthy of encouragement. In order, therefore, to launch the association upon a settled policy in the matter of methods the primal reason for our existence as an association I strongly recommend the appointment of a permanent committee on methods, consisting of nine members, who shall hold office until disqualified by the provisions of the constitution; this committee to consist of three subcommittees corresponding to our present committees A, B, and C. This committee shall receive all reports of referees at least three weeks before the annual meeting of the association, should edit them for presentation to the convention, and after the adoption of new methods or modifications of the old ones, should incorporate

them in our official methods in harmony with our newly established system of treatment. In this way the policy of the association as regards methods would continue unaltered year after year, and the committees would not labor under the disadvantage, so often evidenced in the past, of unfamiliarity with the work of their predecessors.

One more presidential suggestion, and I am through. Our constitution provides in definite terms who are eligible to membership in this association. Section 2 reads as follows: "Analytical chemists connected with the United States Department of Agriculture, or with any State or National agricultural experiment station or agricultural college, or with any State or National institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership." And later in the same section we read: "All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote." It is not entirely clear in my mind just what the words "any State or National" mean. I had supposed that our membership was limited to agricultural chemists in this country, but the above wording, according to my interpretation, might include those connected with agricultural institutions in any State or country. It hardly seems possible that this could have been the meaning of the framers of our constitution, and but for the fact that our Canadian confrères have always enjoyed all the privileges of the association, I would not raise the point at this time. We all appreciate the valuable contributions to our methods from our Canadian friends, and I think no one would deny to them the privileges of membership, and yet I believe we should maintain this as a North American association. I would suggest, therefore, that this ambiguity in the constitution be considered at this meeting by a special committee, and would recommend that the constitution be so amended as to include Canada and Mexico as the only foreign countries entitled to representation.

The constitution provides that unofficial analytical chemists are not "entitled to enter motions or vote." Surely the more important duty of acting as referee should also be denied them, and yet they are not specifically barred from this privilege by the constitution. Unofficial chemists have frequently acted as our referees in the past, and in many cases the association has benefited by their labors; and yet, with all respect to these gentlemen, the precedent established is a dangerous one and can not be too speedily corrected. We should heed Virgil's warning: "Timeo Danaos et dona ferentes."

I would not have the members of the association assume from the somewhat critical tone of my remarks that I am unmindful of the excellent work of the past, of the authoritative position the association now occupies in scientific and legal circles, and of the patient, self-denying, self-effacing work of many of our members in advancing agricultural analysis to its present high plane. But self-complacency and self-congratulation are not incentives to determined, progressive action. Much of our work in the past has had to do with the comparatively simple problems of agricultural analysis; the work of the future will demand effort of an even higher grade, based on careful, painstaking, intelligently directed research. Every station owes it to the public to do some work of original investigation. The analytical methods employed are in a sense the tools by which any agricultural chemical investigation is made possible. The perfection of these tools alone makes possible successful investigation with them. It is certainly, then, no unworthy or unimportant work for a station to spend time in the careful study and perfection of methods.

APPOINTMENT OF COMMITTEES.

At the close of the president's address, which was received with marked approval by the association, the following committees were announced:

Committee on resolutions: Messrs. Van Slyke, Hopkins, and Withers.

Committee on nominations: Messrs. Hartwell, Bartlett, and Cathcart.

Committee on amendments to the constitution: Messrs. Frear, Kilgore, and Lipman.

On motion by Doctor Wiley the recommendations offered in the president's address were referred to the committee on resolutions.

The report of the referee on nitrogen was then presented by the secretary as follows:

REPORT ON THE DETERMINATION OF NITROGEN.

By CHARLES L. PENNY, *Referee*.

The following circular letter, together with samples of corn meal and cotton-seed meal, was sent to members of the association:

The referee and associate referee on nitrogen request you to determine the nitrogen and moisture in a sample of corn meal and one of cotton-seed meal sent to you by mail.

It is requested that nitrogen be determined by the Kjeldahl method and also, if convenient, by the soda-lime method or the absolute method. As a comparison of analytical figures is contemplated, with a view to repetition in cases of wide variation from the average, it is hoped that the work may be done promptly.

Please state in reference to the moisture and the Kjeldahl method—

(1) Temperature at which moisture is determined, and also whether in air or hydrogen.

(2) Duplicates of nitrogen separately, not merely the average.

(3) Whether at end of digestion the hot sulphuric acid was quite colorless.

(4) Whether permanganate of potash was used or not; and if used, whether to the point of permanent coloration.

(5) Any other reagents used in the digestion.

(6) The quantity of substance taken for a determination, the amount of sulphuric acid used, and the size of digestion flasks.

(7) The approximate time of digestion and of distillation.

(8) Whether blanks were digested and distilled; and if so, the amount of correction; also whether the distillation was proved to be complete by the collection of a second portion of distillate.

(9) Method of standardizing acid and the indicator used.

It is hoped that all chemists will cooperate in this work, as it is believed that no single determination interests so many as does that of nitrogen, that few are so important, and that unfortunately variations in analytical results have been unreasonably wide.

Address replies to the referee—

C. L. PENNY,
Agricultural Experiment Station, Newark, Del.

FEBRUARY 16, 1907.

Fifty-three analysts took part in the work, contributing a valuable collection of opinions and experiments. The generous portion of time and labor given by them shows a deep interest in every attempt at the improvement of our analytical methods. Over 420 separate determinations of nitrogen and about 120 of moisture were made, some according to strictly prescribed methods and others according to methods deviating in several respects from these. The list of analysts participating in this work may be found in the table of moisture

determinations. The samples were taken from a quantity of material thoroughly mixed, bottled, and sealed; it is believed that they were as nearly uniform as they could be made, and fully secured from change in moisture content or other change. The following table shows the percentage of moisture found by the several analysts and the methods pursued by each:

TABLE I.—*Results of cooperative work on the determination of moisture in corn meal and cotton-seed meal.*

Analyst.	Corn meal.		Cotton-seed meal.		Method.
	Results.	Average.	Results.	Average.	
	Per cent.	Per cent.	Per cent.	Per cent.	
Asbury, S. E.			7.75		Air; 99°-100° C.
Atkinson, F. C.	{ 13.45 13.45 }	13.45	{ 7.60 7.75 }	7.70	
Bailey, E. Monroe					Vacuum; three and one-half hours; 70°-75° C., 2 grams of substance. Thirty-six hours; 96° C. (water). Twelve hours; 100° C. (air).
Barber, Kate E.					
Blair, A. W.	{ 13.42 13.35 }	13.39	{ 7.70 7.85 }	7.78	
Bradley, C. E.	{ 13.55 13.52 }	13.54	{ 7.77 7.77 }	7.77	
Do	{ 14.41 14.43 }	14.42	{ 8.69 8.95 }	8.82	
Carlyle, E. C.					Air bath; 212° F.; five hours.
Crocker, C. S.	{ 14.15 13.87 }	14.15	{ 8.00 7.67 }	8.00	
Cruse, J. C.	{ 13.62 13.87 }	13.75	{ 7.67 7.91 }	7.79	Hydrogen; temperature boiling water: Corn meal—2.981 grams, 2.847 grams; cotton-seed meal—3.071 grams, 2.436 grams.
Edmond, H. D.	{ 15.19 15.15 }	15.17	{ 8.98 9.04 }	9.01	
Fetzer, L. W.	{ 13.34 13.42 }	13.38	{ 8.39 8.41 }	8.40	Air; boiling water oven.
Ford, A. G.	{ 12.14 12.17 }	12.16	{ 6.71 6.60 }	6.66	
Fuller, F. D.	{ 13.15 13.10 }	13.13	{ 7.48 7.47 }	7.47	Air; 98° C.; five hours; 2 grams substance.
Fulmer, H. L.	{ 13.94 13.97 13.93 }	13.95	{ 8.05 8.04 8.18 }	8.09	
Gascoyne, W. J.					Air; water oven; 98° C.; five hours.
Grainger, W. E.	{ 13.18 13.03 }		{ 7.64 7.63 }	7.64	
Do	{ 12.21 12.16 }	12.65			Air; water oven; to constant weight.
Greaves, J. E.	{ 12.67 12.45 }	12.56	{ 6.13 6.15 }	6.14	
Green, H. L.					Air; temperature boiling water; ten hours; 2 grams substance.
Hall, J. A., jr.	{ 12.45 12.50 }	12.48	{ 6.80 6.90 }	6.85	
Halligan, J. E.					Hydrogen; 96° C.
Hammond, H. S.					
Hare, R. F.	{ 13.13 13.53 }	13.33	{ 7.38 7.00 }	7.19	Air; 100°-101° C.
Jones, J. Shirley	{ 14.88 15.10 }	14.99	{ 9.12 9.08 }	9.10	
Kerr, Robert H.	{ 13.03 13.22 }	13.03	{ 7.24 7.46 }	7.24	Air; water oven; five hours.
Knisely, A. L.	{ 13.12 13.69 }	13.17	{ 7.49 7.91 }	7.48	
Do	{ 13.69 13.69 13.96 }	13.69	{ 7.91 7.92 7.36 }	7.92	Air; oven; eighteen hours; 94°-96° C.
Kunst, F. B.	{ 14.25 13.97 }	13.96	{ 7.34 8.40 }	7.35	
La Shell, L. L.	{ 14.19 14.23 }	14.22	{ 8.43 8.45 }	8.43	Vacuo-steam bath; temperature of boiling water; five hours; 2 grams.
Macy, E. J.	{ 13.70 12.87 }	13.70	{ 7.36 7.23 }	7.05	
Do	{ 13.70 13.70 }	13.70	{ 7.54 7.55 }	7.23	Water oven; ten hours.
McDonnell, C. C.	{ 13.38 13.70 }	13.54	{ 7.62 7.75 }	7.55	
Mitchell, S. R.	{ 12.08 12.49 }	12.29	{ 7.07 6.72 }	7.69	Air; 96° C.
Morgan, J. F.	{ 13.65 13.71 }	13.68	{ 7.85 7.83 }	7.69	
Do	{ 14.30 14.35 }	14.33	{ 8.36 8.36 }	6.90	Hydrogen; 96° C.
Do					
					Hydrogen; five hours; 2 grams; steam.
					Hydrogen; ten hours; 2 grams.
					Hydrogen; thirteen hours; 2 grams.

TABLE I.—Results of cooperative work, etc.—Continued.

Analyst.	Corn meal.		Cotton-seed meal.		Method.
	Results.	Average.	Results.	Average.	
	Per cent.	Per cent.	Per cent.	Per cent.	
Morgan, J. F.	{ 14.31 14.39	14.35	{ 8.41 8.39	8.40	Hydrogen; sixteen hours; 2 grams.
Do.	14.36	14.36	{ 8.81 8.70	8.76	{ Paraffin oven; twenty hours; 2 grams; 95° C.
Do.	14.36	14.36	{ 8.86 8.71	8.79	{ Paraffin oven; twenty-three hours; 2 grams; 95° C.
Norton, J. H.	{ 13.03 12.96	13.00	{ 7.64 7.30	7.47	Air; 110° C.; two hours.
Do.			{ 7.36 7.70	7.53	{ Air; 110° C.; three and one-half hours.
Ogden, A. W.	13.07	13.07	7.42	7.42	Air; 100° C.; three hours.
Do.	13.07	13.07	7.47	7.47	Air; 100° C.; five hours.
Do.	13.13	13.13	7.43	7.43	Air; 100° C.; two hours.
Do.	13.15	13.15	7.27	7.27	Air; 100° C.; two and one-half hours.
Do.	15.16	15.16	8.75	8.75	Hydrogen; 100° C.; two hours.
Do.	15.30	15.30	9.04	9.04	Hydrogen; 100° C.; four hours.
Do.	15.06	15.06	8.63	8.63	Hydrogen; 100° C.; three hours.
Pappe, T. F.	{ 11.88 11.79	11.84	{ 6.63 6.63	6.63	{ Air; water oven; temperature of boiling water.
Parkens, J. H.					
Patten, A. J.	{ 13.45 13.43	13.44	{ 7.81 7.67	7.74	
Rather, J. B.					
Robb, J. B.	{ 11.96 11.94	11.95	{ 6.80 6.92	6.86	Air; five hours.
Do.	12.53	12.49	7.17	7.13	Hydrogen; five hours.
Do.	12.44		7.08		
Do.	13.52	13.48	7.75	7.73	Air; twenty-one hours.
Do.	13.43		7.70		
Roberts, George.					
Shanley, E. J.	{ 14.99 15.04	15.02	{ 9.41 9.51	9.46	{ Hydrogen; temperature of boiling water.
Shedd, O. M.	12.79	12.70	7.59	7.52	Air; 96½° C.; five hours.
Do.	12.61		7.44		
Do.	12.79	12.77	7.59	7.54	Air; 96½° C.; to constant weight.
Do.	12.75		7.48		
Do.	14.41	14.43	8.65	8.71	Natural gas; water oven 96½° C.; five hours.
Do.	14.44		8.76		
Do.	14.68	14.70	8.93	8.93	Natural gas; water oven; 96½° C. to constant weight.
Do.	14.71		8.92		
Sheib, S. H.	13.32	13.32	7.55	7.55	Air; water bath; five hours.
Smith, P. H.	14.02	13.99	8.30	8.25	Air; 100° C.; nineteen hours.
Do.	13.95		8.19		
Spears, H. D.					
Summers, J. C.	{ 12.65 12.68	12.67	{ 6.94 7.06	7.00	Air; water oven; 98° C.; eight hours.
Taggart, W. G.					
Thatcher, R. W.	{ 14.66 14.68	14.67	{ 9.13 8.92	9.03	{ Hydrogen; Caldwell tubes; eight hours.
Treacot, T. C.	14.93	14.93	8.78	8.78	Vacuum oven; 96° C.; nine hours.
Watkins, H. R.	{ 14.04 13.97	14.01	{ 8.18 8.17	8.18	Water oven; twelve hours.
Do.			8.54		
Whittier, A. C.			8.43	8.43	Air; 97° C.
Do.			8.33		
Wiley, R. C.	{ 11.77 11.70	11.74	{ 6.72 6.73	6.73	Air; water oven; 100° C.
Williamson, C. S.	12.80	12.80	7.38	7.38	Air; 100° C.; four hours.
Wilson, R. N.	{ 13.23 13.22	13.23	{ 7.10 7.16	7.13	{ Vacuum; 70°-75° C.; three and one- hours; 2 grams.
Mean of all different determinations.		13.54		7.82	
Maximum		15.30		9.46	
Minimum		11.74		6.14	

The moisture was determined in a variety of ways, and there is considerable divergence in the results, partly due to difference in the method and partly to the analysts, but, it is believed, due only slightly to actual differences among the samples. The fluctuations in the moisture of the corn meal, 3.56 per cent, are nearly one-fourth and in that of the cotton-seed meal, 3.332 per cent, over one-third of the actual moisture in the respective substances.

Two well-known and important facts appear from the comparative work of some of the analysts, namely, the oxidizing action of the air, which sometimes accounts for a loss of over 2 per cent of moisture as compared with a determination in hydrogen and the great influence exerted by the length of time allowed for drying. While it may not, perhaps, be within the scope of a report on determining nitrogen, I would emphasize the advantage of returning to a four or five hour time limit for moisture determinations in feeds and similar products. The figures of Mr. Morgan and Mr. Knisely show that even ten or eighteen hours of heating do not give constant weight. Probably every feed control laboratory establishes its own arbitrary time limit, but it would be better if a fixed temperature and a time limit were prescribed to provide against variations of the boiling point at different altitudes.

The following table shows the percentages of nitrogen as determined by 52 analysts, together with the various methods pursued. The numbers assigned are arbitrary and do not refer to the preceding table:

TABLE II.—Percentages of nitrogen found in corn meal and cotton-seed meal, based on the air-dry, not the absolutely dry, state.

(a) SULPHURIC ACID METHODS.

Number of analyst.	Nitrogen.				Description of method.
	Corn meal.		Cotton-seed meal.		
	Per cent.	Per cent.	Per cent.	Per cent.	
1.....	1.475 1.465	1.47	6.797 6.800	6.799	{ Digested two and one-half hours; colorless; green with KMnO_4 ; 800 cc digestion flasks.
2.....	1.450 1.410 1.412	1.424			{ Same as above except one and one-half hours' digestion.
	1.450 1.490 1.470	1.467	6.71 6.72 6.79	6.74	{ Same as above except one hour digestion.
3.....	1.471 1.414 1.465	1.443	6.71 6.71	6.71	{ Same as above except two and one-half hours' digestion.
	1.472	1.469			{ Same as above except three hours' digestion.
4.....	1.41 1.42 1.40	1.41	6.70 6.69 6.65	6.68	{ Gunning; 20 cc H_2SO_4 ; 10 grams K_2SO_4 ; digested four hours.
5.....	1.38 1.40	1.39	6.67 6.67	6.67	{ Gunning; with 0.1 gram copper sulphate; one hour digestion.
6.....	1.43 1.43	1.43	6.70 6.62 6.67	6.66	{ Mercuric oxid; colorless; green with KMnO_4 ; one and one-fourth hours' digestion.
			6.72 6.83	6.775	{ Same as above except three hours' digestion.
7.....	1.51 1.48	1.495	6.85 6.85	6.85	{ Mercury; colorless; green with KMnO_4 ; five hours' digestion; distilled in current of steam.
8.....	1.45 1.43 1.47 1.47 1.47	1.44 1.47 1.47	6.75 6.75 6.77 6.79 6.83	6.75 6.78 6.83	{ Mercury; three hours' digestion; no KMnO_4 .
					{ The same as the last, except KMnO_4 to permanent coloration.
					{ The same, except five hours' digestion and no KMnO_4 .
	1.47 1.46 1.465 1.465	1.465 1.465 1.465	6.83 6.83 6.84 6.82	6.83 6.84 6.82	{ Mercury; 10 grams K_2SO_4 ; three hours' digestion; no KMnO_4 .
					{ The same, except five hours' digestion.
					{ The same as the last, except KMnO_4 to permanent coloration.
9.....	1.453 1.460 1.453	1.455			{ 12 grams K_2SO_4 ; 0.5 gram Hg; no KMnO_4 ; two and one-fourth hours' digestion.
			6.873 6.873 6.852 6.860	6.866	{ The same, except three hours' digestion.
10.....	1.442 1.442 1.435 1.435 1.456 1.456 1.449 1.449	1.439 1.453	6.839 6.846 6.881 6.830 6.818 6.860	6.857 6.836	{ Official Kjeldahl; KMnO_4 used; one hour digestion.
			6.965 6.958	6.962	{ Kjeldahl-Gunning; Hg but no KMnO_4 ; two-thirds hour digestion.
					{ Same as last, except one hour digestion.

TABLE II.—Percentages of nitrogen found in corn meal, etc.—Continued.

Number of analyst.	Nitrogen.				Description of method.
	Corn meal.		Cotton-seed meal.		
	Per cent.	Per cent.	Per cent.	Per cent.	
11.....	1.50 1.44	1.47	6.78 6.54	6.66	{Hg; one and one-sixth hours' digestion; KMnO_4 .
12.....	1.49 1.43	1.46	6.60 6.60	6.45	{Same as above, except digested till colorless; time not given.
13.....	1.43 1.46 1.40	1.43	6.74 6.76	6.75	Same as above, but time not given.
14.....	1.46 1.46 1.50 1.50	1.47	6.60 6.64 6.68	6.64	Same as last.
15.....	1.50 1.52	1.51	6.80 6.80 6.84	6.81	{Kjeldahl; one and one-fourth hours' digestion till colorless; KMnO_4 .
	1.54 1.56	1.55			{Gunning; No KMnO_4 ; digested two hours till colorless.
	1.42 1.44	1.43	6.82 6.80	6.81	{Kjeldahl-Gunning; digested three-fourths of an hour till colorless; no KMnO_4 .
			6.74 6.70	6.72	{Kjeldahl but no KMnO_4 ; digested one and one-fourth hours till colorless.
			6.88 6.88 6.92 6.92 6.86 6.86	6.89	{Kjeldahl with KMnO_4 ; three hours' digestion.
16.....	1.49 1.46	1.475	6.82 6.86	6.84	{Gunning; two and one-half hours' digestion.
17.....	1.47 1.49 1.46	1.48 1.46	6.84 6.88	6.86	{Kjeldahl; HgO and KMnO_4 .
	1.44	1.44			{Hg; 10 grams K_2SO_4 ; no permanganate; two hours' digestion; colorless.
			6.87	6.87	Same, except three hours' digestion.
					Same, except nearly colorless, faint yellow; three and one-half hours' digestion.
18.....	1.46 1.46 1.45	1.46	6.85	6.85	Same, except five hours' digestion.
					{Dyer modification; i. e., Gunning with Hg and K_2S ; no KMnO_4 ; one and one-fourth hours' digestion.
			6.83 6.76 6.79 6.85 6.74 6.78 6.79	6.79	Same, except two hours' digestion.
				6.795	Same, except one hour digestion.
				6.785	Same, except three and one-half hours' digestion.
	1.42 1.42 1.44	1.43	6.72 6.74	6.73	{Kjeldahl method; digestion, two hours for corn; two and one-half for cotton seed.
			6.63	6.63	Same, except six and one-half hours' digestion. With Dyer modification solutions were quite colorless; those with Kjeldahl method showed faint color.
19.....	1.43 1.43	1.43	6.58 6.44 6.54	6.52	{ HgO ; no KMnO_4 ; digested two hours; corn was colorless; cotton seed pale straw color.
20.....	1.40 1.40 1.42	1.41	6.49 6.49 6.50	6.49	Same as last, except digestion fifty minutes.
21.....	1.416 1.429	1.423	6.666 6.462	6.564	{Kjeldahl; Hg; solutions nearly colorless with a slight yellowish tint in some cases; KMnO_4 to permanent coloration.
22.....	1.43 1.46 1.43	1.44	6.37 6.34	6.355	{Hg; digestion seventy minutes for corn and one hundred and ten minutes for cotton seed; hot solution of corn was colorless, of cotton seed very pale straw color; KMnO_4 added to permanent coloration.
23.....	1.49 1.52	1.505	6.49 6.49	6.49	{Gunning; K_2SO_4 and H_2SO_4 only; six hours' digestion; solution practically colorless.

TABLE II.—Percentages of nitrogen found in corn meal, etc.—Continued.

Number of analyst.	Nitrogen.				Description of method.
	Corn meal.		Cotton-seed meal.		
	Per cent.	Per cent.	Per cent.	Per cent.	
24.....	1.40	1.41	6.65	6.643	{ Kjeldahl; Hg; no KMnO_4 ; digested one and one-half to two hours till colorless.
	1.42		6.64		
	1.40		6.64		
	1.42				
25.....	1.45	1.45	6.82	6.84	{ Kjeldahl; HgO ; digested three and one-half to four hours; colorless with corn, and straw color with cotton seed.
	1.45		6.87		
	1.44		6.84		
	1.44	1.44	6.68	6.84	{ Same as last, except Hg instead of HgO .
	1.44		6.70		
	1.46		6.75		
26.....	1.389	1.401	6.663	6.684	{ Kjeldahl; five hours' digestion; faint yellow color; KMnO_4 to permanent green.
	1.412		6.705		
27.....	1.398	1.379	6.621	6.629	{ Same as last, except four hours' digestion.
	1.389		6.636		
28.....	1.372	1.400	6.678	6.701	{ Kjeldahl, but no KMnO_4 , and no CuSO_4 .
	1.428		6.734		
			6.692		
			6.650		
	1.442	1.419	6.692	6.678	{ Kjeldahl and 0.2 gram CuSO_4 ; $5\text{H}_2\text{O}$; no KMnO_4 .
	1.400		6.692		
	1.414		6.748		
			6.650		
	1.414	1.421	6.720	6.706	{ Gunning, without CuSO_4 .
	1.428		6.748		
			6.748		
			6.776		
29.....	1.40	1.39	6.68	6.71	{ Kjeldahl; digested one and one-half hours; colorless; KMnO_4 to permanent green.
	1.41		6.68		
	1.39		6.72		
	1.38		6.68		
	1.40		6.70		
	1.38		6.72		
	1.39		6.66		
	1.40		6.74		
	1.39		6.78		
	1.38		6.70		
	1.38		6.70		
	1.39		6.74		
	1.39		6.70		
	1.37		6.74		
	1.38		6.76		
	1.39		6.80		
30.....	1.37	1.39	6.74	6.735	{ Same as above, except digested two and one-half hours.
	1.40		6.74		
	1.42		6.70		
			6.70		
	1.41		6.70		
			6.825		
	1.43		6.765		
	1.39		6.855		
31.....	1.40	1.425	6.83	6.80	{ Colorless; KMnO_4 to permanent coloration.
	1.42		6.73		
	1.455		6.835		
	1.435		6.85		
32.....	1.44	1.444	6.755	6.823	{ Do.
	1.46		6.865		
33.....	1.40	1.41	6.66	6.66	{ Gunning; four hours' digestion.
	1.43		6.66		
	1.40				
	1.43				
	1.43				
34.....	1.43	1.43	6.68	6.68	{ Kjeldahl; four hours' digestion.
	1.43		6.68		
	1.50		6.84		
	1.53		6.84		
35.....	1.53	1.52	6.87	6.85	{ Kjeldahl modified for nitrates; five hours' digestion; not colorless; KMnO_4 to permanent coloration.
36.....	1.48	1.48	6.90	6.89	{ Kjeldahl; Hg; one and one-half to one and three-fourths hours' digestion; colorless; KMnO_4 to permanent coloration.
	1.48		6.88		
37.....	1.50	1.50	6.88	6.88	{ Gunning, with 0.5 gram copper sulphate, 10 grams K_2SO_4 and 0.5 gram Hg; no KMnO_4 ; not strictly colorless; one and three-fourths hours' digestion.
	1.351		6.587		
	1.344		6.608		
	1.365		6.601		
38.....	1.422	1.420	6.867	6.877	{ Kjeldahl; HgO ; digestion two hours and fifty minutes; colorless; KMnO_4 added to permanent pink.
	1.419		6.893		
	1.419		6.871		

TABLE II.—Percentages of nitrogen found in corn meal, etc.—Continued.

Number of analyst.	Nitrogen.				Description of method.
	Corn meal.		Cotton-seed meal.		
	Per cent.	Per cent.	Per cent.	Per cent.	
38.....	{ 1.40 1.44 }	1.42	{ 6.79 6.81 6.81 }	6.803	{ Plain Kjeldahl; Hg; pale yellow color; KMnO ₄ to permanent coloration; one and three-fourths' hours digestion.
	{ 1.46 1.46 }	1.46	{ 6.90 6.85 6.87 }	6.873	{ Kjeldahl modified for nitrates; Hg; KMnO ₄ as above.
39.....	{ 1.650 1.651 1.612 1.629 1.635 1.659 }	1.639	{ 7.146 7.201 7.219 7.114 7.179 7.121 }	7.163	{ Kjeldahl; HgO; colorless; KMnO ₄ to permanent coloration; digestion, corn three hours, cotton seed four and one-half hours.
40.....	{ 1.44 1.39 1.41 1.43 1.44 1.42 }	1.42	{ 6.84 6.85 6.82 }	6.837	{ Gunning; no KMnO ₄ ; two hours' digestion.
41.....	1.48	1.48	6.88	6.88	Kjeldahl; HgO; colorless; KMnO ₄ to permanent coloration; one and one-half hours' digestion.
42.....	1.45	1.45	6.85	6.85	Do.
43.....	1.48	1.48	6.84	6.84	Do.
44.....	{ 1.344 1.316 1.43 }	1.330	{ 6.356 6.342 }	6.349	{ Gunning; straw color; no KMnO ₄ ; two hours' digestion.
	1.42	1.42	6.71	6.71	Hg; colorless; one and one-half to two hours' digestion; no KMnO ₄ ; no K ₂ S.
	1.42	1.42	6.78	6.78	Hg; colorless; KMnO ₄ to green color; no K ₂ S.
45.....	1.45	1.45	{ 6.69 6.76 6.74 6.75 }	6.735	Same as last except K ₂ S was added.
	1.38	1.38	6.68	6.68	Same as last except KMnO ₄ was omitted.
46.....	{ 1.40 1.40 }	1.40	{ 6.60 6.64 }	6.62	{ HgO; slightly yellow; KMnO ₄ to permanent coloration; two to three hours' digestion.
47.....	{ 1.50 1.50 1.39 }	1.50	{ 6.83 6.80 6.68 }	6.815	{ HgO; two hours' digestion; colorless; KMnO ₄ to permanent coloration.
48.....	{ 1.43 1.42 1.44 1.44 1.44 }	1.42	{ 6.76 6.66 6.70 6.67 }	6.70	{ Gunning; K ₂ SO ₄ and CuSO ₄ ; one and one-half hours' digestion; colorless; no KMnO ₄ .
	1.43	1.437	6.80	6.71	{ HgO; colorless; corn three hours and cotton seed five hours' digestion; KMnO ₄ to permanent coloration.
49.....	{ 1.43 1.44 1.43 1.41 1.43 }	1.423	{ 6.66 6.76 6.67 6.82 }	6.75	Same as above except no KMnO ₄ .
	1.43	1.427	6.74	6.747	{ Gunning; three and one-half hours' digestion; colorless; no KMnO ₄ .
50.....	{ 1.44 1.44 1.46 }	1.447	{ 6.75 6.77 6.78 6.70 }	6.767	{ Same as above with the addition of HgO; two hours' digestion.
	1.54	1.52	6.70	6.69	{ Hg; almost colorless; KMnO ₄ to permanent coloration; digestion three and one-fourth to four hours.
51.....	{ 1.50 1.54 }	1.52	{ 6.67 6.65 }	6.66	{ Same as above except that after KMnO ₄ was added acid was boiled again until colorless.
52.....	{ 1.440 1.445 }	1.443	{ 6.73 6.72 }	6.725	{ Hg; four and one-half hours' digestion; KMnO ₄ to permanent coloration.
Maximum.....		1.64		7.163	
Minimum.....		1.33		6.349	
Mean of all separate determinations.....		1.446		6.749	
Mean deviation.....		0.035		0.110	

(b) SODA LIME METHOD.

Number of analyst.	Corn meal.		Cotton-seed meal.	
	Results.	Average.	Results.	Average.
	Per cent.	Per cent.	Per cent.	Per cent.
21	{ 1.734 1.752 }	1.743	{ 6.934 6.855 }	6.895

The mean per cent of nitrogen determined is 1.446 for the corn meal and 6.749 for the cotton-seed meal. The various determinations range from 0.12 per cent below and 0.19 per cent above the mean for corn meal and for the cotton-seed meal 0.40 per cent below and 0.41 per cent above. If the mean of the several determinations be taken as the commercially correct figure, it appears that the average error is 0.035 per cent of nitrogen for corn meal and for cotton-seed meal 0.11 per cent. On the corn meal 41 out of 52 analysts, or 80 per cent, estimated within 0.05 per cent of the mean, and in the case of the cotton-seed meal 29 analysts, or 56 per cent, estimated within 0.10 per cent. This is a most satisfactory result, so far as these particular analysts are concerned, i. e., the 80 per cent and the 56 per cent, respectively. It seems probable that the limits just mentioned, 0.05 per cent for corn meal and 0.10 per cent for cotton-seed meal, above or below the accepted average, are the best that can be expected of a large number of analysts working in different laboratories and following a general description of a method. It would be desirable to make the limits of permissible error narrower, for mistakes are costly; but this represents the present state of accuracy and about one-fifth of our cooperating colleagues fail to attain even this limit in their corn meal determinations and over two-fifths that for cotton-seed meal.

The fact that a large number of analysts do agree closely is a most gratifying improvement over the experience of former years. It is doubtful if any further definition can be made to bring about a closer agreement. The only possible solution, in my opinion, is in concrete standards such as the samples sent out have now come to be.

The errors giving too low results may be explained by (1) a false weight (a most improbable source of error); (2) incomplete digestion, leaving some un-reduced nitrogen; (3) excessive digestion, sometimes attended by the decomposition of ammonium sulphate; (4) mechanical loss from spurting; (5) incomplete distillation; (6) escape of unabsorbed ammonia in distilling, either through loose fittings or an unsubmerged outlet tube; (7) a false standard acid. Errors in excess are harder to explain. They may be due to (1) a false weight; (2) the incrustation of flasks with ammonium chlorid; (3) the carrying over of caustic alkali; (4) a false standard acid; (5) impure reagents; (6) with a glass condenser a possible solution of alkali.

A common fallacy in connection with this subject is that the differences in moisture contribute much to the variation in percentage of nitrogen. But a simple calculation will show that the extreme variations in moisture, as estimated, would cause a difference of 0.06 per cent nitrogen in the corn meal, and of 0.24 per cent in the cotton-seed meal. Furthermore, as it happens, the greater part of the analyses of both samples show that the moisture and nitrogen deviate from the mean in the same direction, i. e., both greater or both less; whereas if variation in moisture caused or contributed to the variation in nitrogen content the two quantities should vary in opposite directions. The only reasonable inference is that personal carelessness or inaptitude causes the widest deviations from the mean. After a consideration of many analyses, therefore, it would seem that 0.05 per cent of nitrogen in corn meal and 0.10 per cent in cotton-seed meal, are fair allowances for deviation. It is believed that the two samples submitted to the association have been analyzed accurately. The one can not be far from 1.45 per cent and the other not far from 6.75 per cent of nitrogen. They are hermetically sealed and nearly enough alike (as regards loss or gain of moisture) to serve as useful standards of comparison. The mean figures may not be absolutely free from error when the sulphuric acid method is used (the Kjeldahl method with its various amend-

ments and modifications), but as this method is followed almost exclusively in commercial work and investigations, they are at least conventionally correct.

Determinations by the soda lime method were made by one analyst, with results considerably higher than the other averages. But the data are so meager that no reliable conclusion can be drawn.

Four recommendations are made. The first one calls for from 20 to 30 cc of sulphuric acid instead of merely 20 cc, on the ground that there is danger of the flame striking the bare wall of the flask and decomposing some of the ammonium sulphate. The third one requires the condenser tube to dip below the surface of the standard acid on the ground that when there is much ammonia some may easily escape unless the tube is submerged. The second and the fourth permit the use of copper sulphate, in both the Kjeldahl method proper and in the Gunning modification of it. The claims made for the catalytic action of copper sulphate seem to be justified by experience, and there is reason to suppose that this reagent will do much to shorten the time of digestion. It is well nigh inconceivable that its presence can be in anyway hurtful, and there seem to be many analysts who are very desirous of using it and at the same time of following strictly to the official methods.

It is recommended that—

(1) Bulletin 107, p. 6, under (3) Determination, third line of first paragraph, be changed from "20 cc" to "20 cc-30 cc" (i. e., of sulphuric acid).

(2) Ibid., line 4, after "acid," add "From 0.1 to 0.3 gram of crystallized copper sulphate may also be used in addition to the mercury, or in lieu of it."

(3) Ibid., p. 6, line 6 from bottom, after "standard acid," add: "During at least the first ten minutes of distillation the lower end of the condenser tube should dip beneath the surface of the standard acid."

(4) Ibid., p. 7, (b) Gunning Method, (3) Determination, line 4, after "sulphuric acid," add: "From 0.1 to 0.3 gram crystallized copper sulphate may also be added."

(5) Ibid., p. 7, (3) Determination, line 4, after "sulphuric acid," write: "Approximately 0.7 gram of mercuric oxid or its equivalent in metallic mercury may also be added, previously to the addition of the potassium sulphate, but if mercury be used potassium sulphid must be used, as in the Kjeldahl method, in the distillation."

A general discussion of the third recommendation made by the referee followed, showing a wide variation in practice and opinion regarding this detail of manipulation. The recommendation made was said to be in harmony with the practice in the Bureau of Chemistry, while Mr. Lipman stated that on a thousand determinations made by the New Jersey station without having the tube dip beneath the acid, almost perfect agreement was obtained. Mr. Patrick and Mr. Haskins spoke of the satisfactory results obtained by using a trap if the procedure recommended by the referee were not followed, and Doctor Wiley and Mr. Hopkins called attention to the bearing of temperature on the question. [It is to be noted that in the case of soils, fertilizers, and other low-grade materials, the variation introduced by this loss of ammonia is much less marked than when such materials as meat, cheese, and milk are under consideration. This recommendation was not adopted when presented to the association by Committee A (see page 129).]

REPORT ON THE SEPARATION OF MEAT PROTEIDS.

By F. C. Cook, *Associate Referee.*

Samples of commercial meat extract and the following letter of instructions were sent to the seven collaborators, who expressed a willingness to cooperate on meat proteids this year:

SEPTEMBER 4, 1907.

DEAR SIR: I am sending you, under separate cover, a sample of meat extract. The sample should be kept cool until ready for work, then opened and well mixed.

DIRECTIONS.

Total nitrogen. (1) Determine the total nitrogen in not over 0.5 gram sample. *Soluble nitrogen.* (2) Dissolve 10 grams of the extract in water and make up to 500 cc. Filter on a plain filter paper, pouring back the first 10 or 20 cc. This is filtrate A. Determine the soluble nitrogen in 25 cc of filtrate A.

For the acidity determinations use two 10 cc portions of filtrate A. In one case use phenolphthalein as indicator, diluting the solution before titrating. In the second case employ delicate litmus paper, testing by taking a drop outside by means of a small capillary tube.

In determining the coagulable proteid take two 50 cc portions of filtrate A.

To No. 1 add 9 grams of sodium chlorid, heat to boiling, add 1 cc N/10 acetic acid and boil three minutes. Let stand on steam bath ten minutes, filter, and wash with 50 cc hot water.

Heat No. 2 to boiling and add 1 cc of N/10 acetic acid, boil three minutes, let stand on steam bath ten minutes, filter, and wash with 50 cc hot water.

In both cases determine the nitrogen on the filter paper. Use nitrogen-free filter paper.

The amido nitrogen is determined by placing 25 cc of filtrate A in a 100 cc flask. Add 15 grams of sodium chlorid and 25 cc of water. Shake and keep cool (15° C. or less) for two or more hours. Make a 24 per cent tannic-acid solution, filter and keep at same temperature. Add 30 cc of the 24 per cent tannic-acid solution to the flask, fill to mark with water, shake, and keep cool (15° C. or less) for twelve hours (usually overnight). Filter off 50 cc of the solution and determine nitrogen therein by adding a few drops of sulphuric acid and evaporating to dryness on the steam bath with help of vacuum if at hand. Add 25 or 30 cc of sulphuric acid but no potassium sulphate and proceed as in the Gunning method. It is necessary to run a blank, as the tannic acid often contains nitrogen.

The nitrogen figure minus the blank nitrogen figure multiplied by 2 equals amido nitrogen in the filtrate from 25 cc of the original sample (0.5 gram).

Kreatinin determination. Coagulate 20 cc of filtrate A, as in No. 2 under coagulable proteid, filter, and wash with hot water. Place the filtrate in a 500 cc flask, add 15 cc of saturated picric acid solution and 5 cc of 10 per cent sodium hydroxid, shake well. After standing 5 minutes dilute to mark with water and compare the color with an N/2 potassium dichromate solution in a Dubosc colorimeter, the scale being set at 8 mm; 81 divided by reading x on the other scale equals milligrams of kreatinin. This is a modification of Folin's method as applied to the estimation of kreatin and kreatinin in the urine.^a For the determination of kreatin use 15 cc of filtrate A, coagulate as above, filter, and wash. Place in an Erlenmeyer flask. Add 5 cc of N/2 hydrochloric acid and attach a reflux condenser, place on steam bath for 3½ hours, cool, transfer to a 500 cc flask, add 5 cc of N/2 sodium hydroxid and proceed as above, adding 15 cc picric acid, etc. This reading gives the total kreatinin. From the total kreatinin subtract the original kreatinin and multiply the difference by 1.16 to obtain the kreatin originally present.

Report results as follows:

Total nitrogen, per cent.
Soluble nitrogen, per cent.
Coagulable nitrogen, per cent.
Amido nitrogen, per cent.
Kreatinin, per cent.
Kreatin, per cent.

^a Zts. physiol. Chem., 1904, 41: 223; Amer. J. Physiol., 1905, 13: 48.

Acidity (1) using litmus cc N/10 sodium hydroxid per 100 grams.

(2) using phenolphthalein cc N/10 sodium hydroxid per 100 grams.

Coagulable nitrogen, using sodium chlorid, per cent.

Duplicate determinations should be made. Any criticisms and suggestions will be gladly received.

DISCUSSION OF RESULTS.

In the sample sent out there was but a small amount of insoluble proteid present, consequently no conclusion can be drawn under that head. The cooperative results as given in Table I show a wide variation for total nitrogen, and consequently for the different nitrogenous constituents, as the nitrogen is the basis of the determination.

TABLE I.—Cooperative work on meat proteids.

Analyst.	Nitrogen found as—					
	Total.	Soluble.	Insoluble by difference.	Coagulable.	Proteose and peptone.	Amido.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Grindley & Mitchell, Urbana, Ill.....	8.84	8.67	0.17	0.01	3.92	4.74
A. W. Dox, Storrs, Conn.....	9.23	9.22	.01	.01	4.13	5.18
Tottingham and Hart, Madison, Wis....	9.60	9.42	.18	.055	3.595	5.77
E. W. Rockwood, Iowa City, Iowa:						
Chemist 1.....	10.20	8.42	1.78	.76	3.77	3.89
Chemist 2.....	10.12	8.23	1.89	.80	4.20	3.23
F. C. Cook, Washington, D. C.....	9.30	9.21	.09	.185	5.095	3.93
P. Rudnick, Chicago, Ill.....	9.40	9.40	.00	.00	4.13	5.27
W. D. Richardson, Chicago, Ill.....	9.68	9.66	.02	.020	6.78	2.88
Lowenstein and Dunne, Chicago, Ill.....	9.20	8.93	.27	.000	3.30	5.63
H. C. Jackson, Albany, N. Y.....	9.71	9.56	.15	.072	3.14	6.35

Analyst.	Kreatin.	Kreatinin.	Acidity (N/10 alkali per 100 grams).		Coagulable proteid-nitrogen, using sodium chlorid.
			Phenolphthalein.	Litmus.	
	<i>Per cent.</i>	<i>Per cent.</i>	<i>cc.</i>	<i>cc.</i>	<i>Per cent.</i>
Grindley & Mitchell, Urbana, Ill.....	2.39	4.02	1242		0.01
A. W. Dox, Storrs, Conn.....			1281	1255	.03
Tottingham and Hart, Madison, Wis.....	.348	7.90	1208	933	.102
E. W. Rockwood, Iowa City, Iowa:					
Chemist 1.....	.85	.80	1697		.66
Chemist 2.....	.45	.85	1697		.64
F. C. Cook, Washington, D. C.....	.371	4.50	1325	1025	.305
P. Rudnick, Chicago, Ill.....	1.38	3.24	1400	975	.000
W. D. Richardson, Chicago, Ill.....	2.41	3.21	1350	1000	.018
Lowenstein and Dunne, Chicago, Ill.....	.51	4.22	1450	1500	.000
H. C. Jackson, Albany, N. Y.....			1850	1238	.102

The tannin-salt method gave fairly satisfactory results, although there is still considerable variation in the results obtained by the different collaborators. The trouble often experienced by the foaming in the Kjeldahl process, due to the presence of the tannin, is eliminated by evaporating the solution, after adding 2 or 3 drops of sulphuric acid, almost to dryness, then adding about 30 cc of sulphuric acid and no potassium sulphate, and digesting as usual. The proteose and peptone figures reported were obtained by difference.

Cooperation was also obtained on the determination of coagulable proteids. There has been considerable discussion as to the use of sodium chlorid for this purpose, and the collaborators were instructed in one case to add 9 grams of sodium chlorid to 50 cc of the meat extract solution, while in the other case no sodium chlorid was to be added. As would be expected, practically all of the

results were higher when sodium chlorid was used, but as the amount of coagulable proteid present in the sample was small the results are not striking.

In Table II some results obtained by the referee are given, showing the influence of varying amounts of sodium chlorid on the coagulation of proteid in a commercial meat juice.

TABLE II.—*Coagulable proteid tests using varying amounts of sodium chlorid and 50 cc of the meat extract solution.*

Num- ber.	Sodium chlorid added.	Nitrogen precipi- tated.	Num- ber.	Sodium chlorid added.	Nitrogen precipi- tated.
	<i>Grams.</i>	<i>Grams.</i>		<i>Grams.</i>	<i>Grams.</i>
1	0	0.00842	6	6	0.01263
2	0	.00870	7	9	.01516
3	3	.01039	8	9	.01516
4	3	.01011	9	18	.01881
5	6	.01263	10	18	.01909

These figures show that the amount of nitrogen precipitated increases with the amount of sodium chlorid used, and Mr. Lowenstein, chemist for Morris & Co., Chicago, has obtained similar results for coagulable proteids on samples of meat extracts. It is evident, therefore, that to obtain correct figures for coagulable proteid nitrogen no sodium chlorid should be employed.

The two indicators, phenolphthalein and litmus, used in determining the acidity of meat products were compared, and the results obtained by the various chemists show that in practically all cases higher results were obtained where phenolphthalein was used. In a good many cases the difference is marked. The reason for this is that many of the organic acids have no action on litmus, but do act on phenolphthalein. The referee finds that phenolphthalein inevitably gives higher results than litmus, is easier to handle, and the end point is more accurate, but in certain dark-colored solutions of meat preparations litmus paper must be used.

In Table III the results of acidity tests on solutions of meat extract and on similar solutions after removing the insoluble proteid by filtering, and after coagulating and filtering, are given:

TABLE III.—*Acidity determinations.*

[Comparing the original solution, the solution after filtering, and the solution after coagulation and filtering, using two indicators.]

Num- ber.	Sample		Indicators (N/10 alkali).	
	Amount.	Description.	Litmus.	Phenolphthalein.
	cc.		cc.	cc.
1	25	Meat extract solution.....	4.20	7.575
2	25	Meat extract solution, filtered.....	2.55	7.350
3	25	Meat extract solution, coagulated and filtered.....	3.35	7.575
4	10	Meat extract solution.....	2.20	2.50
5	10	Meat extract solution, filtered.....	2.00	2.50
6	10	Meat extract solution, coagulated and filtered.....	1.80	2.95
7	20	Commercial beef juice, diluted.....	16.30	
8	20	Commercial beef juice, filtered.....	10.15	
9	20	Commercial beef juice, coagulated and filtered.....	8.50	

As the amount of insoluble proteid present in most of the samples was small, the results on this point are not conclusive, but the two latter sets of figures show lower results than those obtained by titrating the original solution, and in two cases the removal of the coagulable proteid before titration gives the lowest results. This is true only when litmus is used, the phenolphthalein

results showing but little variation among themselves. As the amount of proteid present in meat products affects the acid determination, a uniform procedure should be adopted. In the case of meat extracts, most of which contain little insoluble and coagulable proteid, the titration of the original solution is recommended.

The determination of kreatin and kreatinin was also investigated this year. According to the method suggested for kreatinin 20 cc of the coagulable proteid filtrate are used for the determination, but as this filtrate has previously been treated with 1 cc of N/10 acetic acid and boiled three minutes, the heat and acidity employed may have changed some kreatin to kreatinin, thereby giving too high results, consequently it is better to apply the kreatinin test to the original solution. This is satisfactory in the case of most meat extracts, as they contain little insoluble proteid. The effect of the presence of a considerable amount of proteid on the kreatinin test has not been determined.

The best method of applying to meat products, especially meat extracts, the kreatin test, including an estimation of both kreatin and kreatinin in the form of kreatinin, has not been evolved. There are several points which remain to be settled. Folin originally applied his method to 10 cc of urine. In the method on which cooperation was requested a much larger volume of the solution was used. Undoubtedly the volume has some influence on the results, and this point needs investigation. Hehner^a in a recent article claims that the addition of 15 cc of picric acid is not sufficient, and that, upon adding 25 or 30 cc, higher and more accurate results are obtained. This point was investigated, using a solution of pure kreatin and a meat extract, and the addition of 30 cc of picric acid gave but slightly higher results than when 15 cc were used.

The question of the amount of acid to be added for the conversion of kreatin into kreatinin, as well as the period of heating, are important factors. The referee has experimented with the cooperative method which is given in the letter of instruction, with the method as outlined by Grindley and Wood,^b and that used by Armour & Co., which is as follows:

Make the volume of the solution up to 25 cc, place in a 6-inch porcelain evaporating dish, add one-half its volume of normal hydrochloric acid, and evaporate nearly to dryness on the water bath. Add 25 cc of water and 12.5 cc of normal hydrochloric acid, and repeat the evaporation to dryness. Take up the residue in 25 cc of water, add 15 cc of picric acid and 5 cc of sodium hydroxid, let stand five minutes, and then rinse into a 500 cc volumetric flask.

This method gives good duplicates and is short and simple. With Grindley's method the duplicates were not so good nor the results so high, and the cooperative method gives lower results than either of the two others. It is evident that three and one-half hours' heating on the steam bath is not sufficient to convert all the kreatin to kreatinin. A method using 10 cc of normal hydrochloric acid and heating four hours in a boiling-water bath gives results that are fully as high as those obtained by Armour & Co. While none of the methods for kreatin and kreatinin are as yet entirely satisfactory, the value of these two determinations in analyzing meats and meat products has been generally recognized. Benedict and Myers^c have employed an autoclave for the conversion of kreatin to kreatinin. By this method fifteen minutes suffice for the complete conversion. The referee has this method under investigation and recommends that the study of an official method for kreatin and kreatinin be continued next year.

^a Pharm. J., 1907, 78: 683.

^b J. Biol. Chem., 1907, 3: 49.

^c Amer. J. Physiol., 1907, 18: 397.

One of the important features of this work is the criticism which it calls forth. The comments of the collaborators, whom the referee here takes occasion to thank for their helpful interest in the work, are as follows:

COMMENTS BY ANALYSTS.

H. S. Grindley, of Urbana, Ill., says: First, I do not think that the operation of determining the soluble nitrogen is of sufficient value to justify the additional labor.

Second, I think there is serious objection to the determination of kreatinin in the filtrate obtained after the coagulation of the solutions by heat and acetic acid. It is my opinion that by such an operation kreatin will be converted into kreatinin. Investigations made in this laboratory conclusively prove that the kreatin existing in water extracts of flesh are very readily, in part at least, converted into kreatinin even in the presence of the neutral acidity of the extract of flesh.

Third, the filtration of the solutions is very slow and tedious, and we have found that unless three or four filters are used for each solution there is an error in all determinations upon the filtrate due to the evaporation of the solutions during filtration.

Fourth, since the filtration of a water solution of meat extract is so difficult, it appears that, as far as possible, determinations should be made directly upon the original solution without filtration. Our work demonstrates that the presence of proteids, even in considerable quantity, does not interfere with the kreatin and kreatinin determinations.

Fifth, the acidity of ordinary extracts can be determined as well upon the solution of the extract before as after filtration. However, we have not found any meat extract which contains any considerable quantity of insoluble matter.

Sixth, the titration of the solutions of the extracts when using litmus as an indicator was very unsatisfactory. Men in this laboratory working independently have obtained such results when using litmus as to indicate clearly the unreliability of this indicator. For these reasons, no results of acidity of meat extracts when litmus is used as the indicator are reported.

Seventh, the tannin-salt method worked very nicely as to details of manipulation, and the duplicate results have been very satisfactory. On the other hand, it seems very unfortunate that a reagent which involves such a large correction factor must be employed. Although we have used Kahlebaum's very best tannin, it contained a large quantity of nitrogen.

Arthur Lowenstein, of Chicago, Ill., makes the following suggestions and criticisms regarding the method:

Total and soluble nitrogen: The methods for these determinations are quite satisfactory providing the sample is absolutely uniform, a difficult matter to accomplish with solid extracts containing 24 per cent moisture or less, and when only 0.5 gram of extract is employed an error is apt to be introduced.

Acidity: With the majority of extracts it is preferable to employ phenolphthalein as indicator. The reaction with litmus is not very sharp and with very highly colored extracts, such as are frequently found, the use of an outside indicator is necessary.

Coagulable nitrogen: The extract submitted contained practically no coagulable matter. However, in other samples the addition of sodium chlorid gave a higher result in every case.

Amido nitrogen: The method of evaporation is slow, and equally good results were obtained if 50 cc of the filtrate were used after precipitation with tannic

acid and digested immediately with sulphuric acid. The digestion with sulphuric acid only is extremely slow.

There seems to be quite a difference in the various tannic acids (c. p.) on the market. Some produce a very voluminous precipitate, others comparatively little. No account is taken in the method of the volume (bulk) of this precipitate. If it is considerable (as it might be in some extracts), it would introduce the same error that one finds in some of the present official methods.

Kreatinin: A criticism pertinent at this point, but also applicable to the methods as a whole, is they are not specific enough. I could readily observe in our own laboratory that the personal equation would be quite a factor. As this is the first year's work on this subject, just such points should be brought out. The method for kreatinin states, "take 20 cc of A, coagulate, filter, and wash." As the sample in question contains no coagulable proteid, we made the kreatinin determination both with and without dilution (washing), and found that the volume of the solution is quite a factor. Since the proposed method is a modification of Folin's method, in which he uses approximately 10 cc of urine, we thought that the more concentrated the solution the better would be the results obtained, and our results seem to confirm this view. Consequently we avoided transferring and washing as much as possible. Hehner states that 15 cc of picric were not sufficient for meat extracts, and also gives the amount of picric acid in solutions saturated at different temperatures, and this ought to be mentioned. Time, unfortunately, did not permit us to confirm Hehner's results, and in the work reported the quantities given in the method by the referee were used. I have heard from chemists who used 25 cc of picric acid, however, that they did not get results nearly so high as Hehner reports.

Kreatin: To determine whether the time of heating the solution on the steam bath was a factor, one determination was left on the steam bath for fifteen hours. The results checked very satisfactorily with the three-and-one-half-hour test.

The Grindley-Woods method was not tried because of lack of time, but we would suggest that after converting into kreatinin the sodium hydroxid and picric acid be added to the original Erlenmeyer flask and not transferred to the 500-cc flask until ready to dilute at the end of five minutes.

One of the principal objections to reporting these results was the age of the sample at the time of analysis. From practical experience it is known that extracts change with age, in fact, seem to improve in flavor in the case of solid or paste extracts, and we did not know whether the changes would be appreciable nor how much they would affect the content of meat proteids and bases and the acidity test.

Mr. Holmes C. Jackson, of Albany, N. Y., writes:

(1) As regards the acidity determination, it would seem to me that the 10 cc portions which were required to be used were a somewhat small fraction of the total, since in calculating the final figures (cc N/10 NaOH per 100 grams) it is necessary to multiply the titration figures by 500. Obviously very slight variations in the titration will give large variations in the final results. As stated last year, I am decidedly in favor of phenolphthalein as an indicator as compared with litmus, since, unless one makes his own paper, and neutral paper at that, I can not see how the necessary accuracy can be obtained.

(2) Concerning coagulable proteid, it is suggested that some mechanical feature in the method be introduced so that during the boiling and heating in the steam bath evaporation does not take place. Although not mentioned in the

method sent out, some such scheme was found to be absolutely necessary. I used a simple unjacketed reflux condenser.

(3) As regards the amido nitrogen, it seems to me that the results can not be absolutely correct, and the cooperative work shows that the results obtained by the different analysts are not comparable. There must be a variable factor introduced as the result of the presence of such a volume of precipitate. I therefore question again the accuracy of multiplying by 2 the results obtained from 50 cc of the filtrate from the original 100 cc of the mixture of filtrate and precipitate. In determining this point, it is suggested that the double dilution method used in the determination of lactose in milk be employed or some similar procedure which takes into account the volume of the precipitate.

The first sample sent me was lost after I had made the coagulable nitrogen analysis, and it gave a much higher result than the second sample sent. If the second sample was the same as the first and had been standing about during that time, is it not possible that a change had taken place in the character of the coagulable proteid by which it was transformed into noncoagulable? Might not this help to explain the great variations in the cooperative results for this factor?

Mr. Arthur W. Dox, of Storrs, Conn., says: "The precipitation of coagulable proteid seems to be incomplete. I would suggest that a greater concentration of acid be used and that the precipitate be washed with water that has been slightly acidulated. With sodium chlorid a higher figure is obtained, although the concentration is not sufficient to cause a precipitation of proteoses. If the latter method is adopted, the precipitate should be washed with a solution of sodium chlorid, so that none of it will be redissolved."

Mr. E. W. Rockwood, of Iowa City, Iowa, says: "The principal difficulties encountered were the indistinctness of the end reaction with litmus and in the colorimetric determination of the kreatin and the kreatinin. The latter was very probably due to lack of experience in judging these shades. The work was verified, and I can not explain the discrepancy in the nitrogen on any other basis than that it was not easy to sample accurately a substance with so high a nitrogen content. Both chemists have had experience in Kjeldahl determinations and worked independently."

Some analyses of commercial samples of meat extract by the cooperative methods are given in Table IV, including analyses by Mitchell and Grindley, of the University of Illinois, and W. D. Richardson, of Swift & Co., of Chicago.

TABLE IV.—*Analyses of commercial meat extracts by referee's methods.*

H. H. MITCHELL AND H. S. GRINDLEY.

[Each figure represents the average of three determinations.]

Number of sample.	Nitrogen.		Coagulable proteid precipitated by—		Amido nitrogen.	Kreatinin.	Kreatin.	Acidity determined by phenolphthalein (N/10 NaOH per 100 grams).
	Total.	Soluble.	NaCl and acetic acid.	Acetic acid.				
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>cc.</i>
2278	7.50	7.20	0.10	0.05	4.13	2.25	2.04	1,173.0
2306	9.01	9.02	.10	.03	5.82	5.32	1.60	1,194.0
2307	8.11	7.98	.03	.06	3.89	2.22	2.05	598.0
2308	6.62	6.42	.06	.07	3.27	1.19	.68	1,304.0
2309	9.11	7.96	.01	.01	5.74	3.69	1.28	990.0
" 2330	8.84	8.67	.01	.01	4.74	4.02	2.39	1,242.0

* Beef extract from the Bureau of Chemistry, U. S. Dept. Agr.

TABLE IV.—*Analyses of commercial meat extracts, etc.*—Continued.

W. D. RICHARDSON.

Sample.	Moisture.	Salt.	Ash.	Nitrogen.						Krea- tin.	Krea- tinin.	Acidity as indicated by (N/10 NaOH per 100 grams).	
				Tot- al.	Solu- ble.	Coagulable.		Ami- do.	Lit- mus.			Phen- ol- phta- lein.	
						With salt.	With- out salt.						
"A," Extract of beef.....	P. ct. 19.54	P. ct. 7.04	P. ct. 23.15	P. ct. 8.56	P. ct. 7.90	P. ct. 0.283	P. ct. 0.176	P. ct. 3.65	P. ct. 0.75	P. ct. 1.89	cc. 900	cc. 1,150	
"B," Beef extract..	20.6	15.43	29.97	7.24	7.18	.031	.088	4.35	.99	3.20	900	1,100	
"C," Extract of beef.....	20.36	13.65	26.23	8.39	7.87	.019	.025 { .03	3.59	.66	1.84	850	1,150	
"D," Extract of beef.....	23.62	11.09	26.87	8.11	7.59	.033	.036	4.32	1.13	1.40	850	1,050	
"E," Beef extract..	22.02	13.01	28.19	7.49	7.15	.026	.031	4.37	.39	2.92	1,000	1,150	
"F," Extractum carnis.....	21.07	3.32	18.29	9.98	9.36	.053	.159	4.96	2.07	3.11	1,300	1,500	
"G," Beef fluid.....	49.67	9.00	19.57	4.44	4.43	.024	.026	2.82	.01	1.60	675	925	

RECOMMENDATIONS.

It is recommended—

(1) That the following method for the determination of the acidity of meat products be adopted as a provisional method:

Determine the acidity by titrating a dilute solution of the meat, or meat preparation, with N/10 alkali, using phenolphthalein as indicator. But if the color prevents the use of this indicator, use litmus paper with the understanding that the results are lower than when phenolphthalein is employed.

(2) That the modified tannin-salt method for the separation of proteoses and peptones from the simpler amido bodies be adopted as provisional. The method reads as follows:

Employ 50 cc of the filtrate from the determination of the coagulable proteins, transfer to a 100 cc graduated flask, add 15 grams of sodium chlorid, 10 cc of cold water, shake well and place in the cold at 12° C. (an ice box is a convenient place.) Prepare and filter a 24 per cent solution of pure tannin, and keep at the same temperature, 12° C., also a flask of distilled water. After the temperature of the contents of the flask has reached 12° C. add 30 cc of the tannin solution, shake thoroughly and fill to the mark with cold water, shake again and let stand at 12° C. for twelve hours (over night). Filter off 50 cc at 12° C. and determine the nitrogen therein as follows:

Transfer 50 cc of the tannin-salt filtrate to a Kjeldahl digestion flask and add a few drops of sulphuric acid. Place the flask in the steam bath, connect with the vacuum, and evaporate to dryness. In the digestion process about 30 cc of sulphuric acid are added, but no potassium sulphate. The remainder of the process is carried out in the usual manner.

A blank must be run simultaneously. The nitrogen in 50 cc of the filtrate above, minus the nitrogen in the 50 cc of the blank filtrate multiplied by 2, gives amido nitrogen. The nitrogen of the proteoses and peptones is determined by difference.

(3) That, inasmuch as the best modification of the Folin colorimetric method for the determination of kreatin and kreatinin in meat products has not been determined upon, this point be studied another year.

(4) That the referee for 1908 investigate the xanthin base method and a method for estimating organic phosphorus.

REPORT ON THE SEPARATION OF VEGETABLE PROTEIDS.

By H. SNYDER, *Associate Referee.*

The work on the separation of vegetable proteids has been confined to a study of the literature on the subject. No attempt has been made to correlate the literature, and only a general summary is given. No samples were distributed for the cooperative work, as previous investigations have shown that in the case of cereals, as in wheat, closely agreeing results can be secured when skilled analysts work on the same sample and follow a definite course of procedure.

There is such an extensive overlapping of the proteids as to solubility that it is doubtful whether accurate analytical methods can be devised for the absolute determination of any one proteid. The water-soluble, dilute salt-soluble, and alcoholic-soluble proteids can be determined with a reasonable degree of accuracy, but each solvent has a tendency to yield a mechanical mixture rather than an individual proteid. In the case of some of the cereals, however, a solvent may extract an individual proteid with a high degree of purity.

In the studies that are being made of the synthetic and ultimate composition of proteids, pure proteids are secured by special treatment not admissible in quantitative work. The object is to secure a pure proteid, and the mechanical losses due to the overlapping of the solvent can be disregarded.

At the present time it would seem best, in the determination of vegetable proteids, to designate the products as water-soluble, salt-soluble, or alcohol-soluble proteids, designating the proteid by the solvent rather than by the specific names suggesting individual proteids, except in special cases, as in wheat, where the alcohol-soluble proteid is mainly gliadin.

There is much need of an improved method for the determination of proteid and nonproteid nitrogen. While the determination of total nitrogen has reached a high degree of perfection, that for the determination of proteid nitrogen is defective. Stutzer's albuminoid nitrogen method does not make a sharp distinction between proteid and nonproteid nitrogen in many substances.

Instances occur where the copper proteid is soluble owing to secondary reactions. In the presence of a large amount of starch the filtration and washing of the precipitate often require hours, rendering the accuracy of the results questionable.

It would seem best first to direct attention to the improvement of the methods for the determination of proteid and nonproteid nitrogen rather than of individual proteids.

The analytical and synthetical work that is being done bids fair to give, in the near future, important results, and then it will be possible to devise rational methods for the separate determination of the various component proteids. To analyze accurately a complex compound, its composition must first be determined.

It is therefore recommended that the referee on vegetable proteids give special attention to the study of methods for the determination of proteid and nonproteid nitrogen.

The association adjourned to meet at 2.30 p. m.

THURSDAY—AFTERNOON SESSION.

On the suggestion of Doctor Frear, the recommendations in the president's address calling for changes in the constitution were referred to the committee on amendments to the constitution for recommendations.

In the absence of Mr. Woll, Mr. Bartlett, the associate referee, presented the report on dairy products.

REPORT ON DAIRY PRODUCTS.

By F. W. WOLL, *Referee*.

The referee was requested, by a vote of the association, to continue the study of the analysis of condensed milk with special reference to the determination of lactose, sucrose, and fat in the sweetened product.

The following explanatory letter, together with two carefully prepared samples of sweetened and unsweetened condensed milk, were sent to 22 different stations where chemists had signified their willingness to cooperate in the work outlined by the referee.

MADISON, WIS., March 28, 1907.

DEAR SIR: As stated in my circular letter of the 12th instant, the cooperative work on dairy products, Association of Official Agricultural Chemists, for this year will be along two lines:

I. *Analysis of condensed milk*, with special reference to the determination of sucrose and lactose in the sweetened product.

The provisional methods for analysis of condensed milk adopted tentatively at the last convention of the association are as follows:

Preparation of sample: Mix thoroughly by transferring the contents of a can to a large evaporating dish and stirring with a pestle. Weigh 40 grams into a 100 cc flask, transfer thereto by washing, and make up to the mark with water.

Total solids: Dilute a measured portion of the above 40 per cent solution with an equal amount of water, use 5 cc of the diluted mixture, and proceed as in the case of milk analysis.

Ash: Ignite the residue from the total solids, cool, and weigh.

Protein: Determine nitrogen by the Kjeldahl or Gunning method in 5 cc of the 40 per cent solution and multiply it by 6.25.

Lactose: Dilute 5 cc of the 40 per cent solution to about 40 cc and add 0.6 cc of Fehling's copper solution; nearly neutralize with sodium hydrate solution and make up to 100 cc. Filter through a dry filter and determine the lactose in an aliquot by the gravimetric and polariscope methods.

Sucrose: Determine (a) gravimetrically and (b) by difference, by deducting the milk solids (lactose, protein, fat, ash) from the total solids.

Fat: Determine by (a) double-extraction method (Circular 32, page 6, Bureau of Chemistry, U. S. Department of Agriculture) and (b) Babcock centrifugal method, using either Leach's modification (Food Inspection and Analysis, page 49) or Farrington's (Wisconsin Station Report 17, page 86).

II. *The Gottlieb method for the determination of fat in milk.*

The directions for the Gottlieb method are as follows (Landw. Vers. Sta., 1892, 40: 1-27):

Ten cc of milk are measured into a glass cylinder $\frac{1}{4}$ inch in diameter and about 14 inches long (see Land. Vers. Sta., 40: 6; a 100 cc burette or a eudiometer tube will do); 1 cc concentrated ammonia is added and mixed thoroughly with the milk. The following chemicals are next added in the order given: 10 cc of 92 per cent alcohol, 25 cc of washed ether, and 25 cc petroleum ether (boiling point below 80° C.), the cylinder being closed with a moistened cork stopper and the contents shaken several times after the addition of each. The cylinder is then left standing for six hours or more. The clear fat solution is next pipetted off into a small weighed flask by means of a siphon drawn to a fine point (see Fig. 6, loc. cit.), which is lowered into the fat solution to within 0.5 cm of the turbid bottom layer. After evaporating the ether solution in a hood, the flasks are dried in a steam oven for two to three hours and weighed. This method is applicable to new milk, skim milk, buttermilk, whey, cream, cheese, condensed milk, and milk powder, but has been found of special value for determining fat in skim milk, buttermilk, cheese, and condensed milk. In the case of products high in fat a second treatment with 10 cc each of ether and petroleum ether is advisable in order to recover the last trace of fat.

Chemists are asked to make comparative fat determinations in a number of samples of skim milk and buttermilk by this and the official method of fat determination in milk. * * *

The referee is indebted to the Borden Condensed Milk Company, New York City, N. Y., who furnished the samples of milk, together with what they believe to be "fair average analyses" of the two products.

TABLE I.—Results as reported by Borden Condensed Milk Company.

Name and number of milk.	Cane sugar.	Milk sugar.	Water.	Protein.	Fat.	Ash.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Sweetened, No. 9.....	43.86	12.08	25.47	8.06	8.75	1.78
Unsweetened, No. 123.....		12.24	70.81	6.97	8.20	1.36

Eight reports of the work done on these samples were received. The results of the analyses given in the following pages have been arranged for the sake of convenience under three headings: (1) Solids, protein, and ash in condensed milk. (2) Fat in condensed milk. (3) Sugar in condensed milk.

SOLIDS, PROTEIN, AND ASH IN CONDENSED MILK.

TABLE II.—Solids, protein, and ash in sample No. 9, sweetened condensed milk.

Analyst.	Solids.	Protein.	Ash.	Analyst.	Solids.	Protein.	Ash.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
J. M. Barnhart, Illinois..	74.32	7.59	1.46	G. E. Patrick and M. Boyle, Washington, D. C.	73.95	8.08	1.67
James M. Doran, Washington, D. C.	74.83	8.38	1.50	P. H. Smith and E. B. Holland, Massachusetts	72.75	7.77	1.71
J. A. Hummel, Minnesota.....	75.33	7.49	1.77	A. C. Whittier, Maine...	73.34	7.53	1.66
Geo. A. Olson, Wisconsin.....	72.02	7.13	1.47				

TABLE III.—Solids, protein, and ash in sample No. 123, unsweetened condensed milk.

Analyst.	Solids.	Protein.	Ash.	Analyst.	Solids.	Protein.	Ash.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
J. M. Barnhart, Illinois..	29.55	7.32	1.34	G. E. Patrick and M. Boyle, Washington, D. C.	29.55	7.69	1.75
James M. Doran, Washington, D. C.	29.81	8.44	1.48	P. H. Smith and E. B. Holland, Massachusetts	29.22	7.49	1.71
J. A. Hummel, Minnesota.....	30.04	7.04	1.79	A. C. Whittier, Maine...	28.58	7.50	1.87
Geo. A. Olson, Wisconsin.....	28.33	7.50	2.17				

^a N×6.38.

REMARKS BY ANALYSTS.

G. E. Patrick: The solids obtained by drying a milk of any kind will vary from 0.1 to 0.2 per cent, according to the method of drying, i. e., whether dried rapidly or slowly, at high or low temperature. In the work here reported samples were dried first on the steam bath and then in boiling-water oven, at atmospheric pressure.

P. H. Smith and E. B. Holland: Moisture: Dried 15 cc of a 20 per cent solution on quartz sand in a flat-bottomed dish at a low temperature until bulk of

the water was expelled, then completed at 100°C. Ash: Evaporated to dryness 25 cc in a platinum dish with 5 cc of concentrated nitric acid and burned to white ash. Protein: Employed the Kjeldahl-Gunning method, using 5 cc of solution.

James M. Doran: Solids: Determined according to the provisional method of the association, evaporating first over steam bath and later drying in water oven to constant weight. Protein: Determined nitrogen in aliquot of diluted milk (corresponding to about two grams of the original sample) by the Gunning method.

Geo. A. Olson: Solids: Determined in the Babcock asbestos tube until constant in weight. Protein: Determined by the straight Kjeldahl method. Ash: Milk first dried in steam oven and finally incinerated in muffle furnace.

DISCUSSION OF RESULTS.

The results for protein and ash on the whole are fairly concordant. This, however, can not be said of those for solids. The difference in solids alone—3 per cent—is sufficient to throw the analyses of the various ingredients out of balance.

FAT IN CONDENSED MILK.

TABLE IV.—*Fat in sample No. 9, sweetened condensed milk.*

Analyst.	Asbestos method.			Babcock test.		Gottlieb method.
	First extraction.	Second extraction.	Total.	Leach.	Farrington.	
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
J. M. Barnhart, Illinois.....			7.78	8.1		
James M. Doran, Washington, D. C.....						9.24
J. A. Hummel, Minnesota.....				7.5		
Geo. A. Olson, Wisconsin.....	3.10	4.56	7.66	8.05	7.75	7.87
G. E. Patrick and M. Boyle, Washington, D. C.....	8.44	0.08	a 8.52			b 8.61
P. H. Smith and E. B. Holland, Massachusetts.....	5.38	2.14	c 7.52			
A. C. Whittier, Maine.....			7.90	8.1		8.15

* Adams paper coll method, 20 per cent solution, first extraction, 8.44; second, 0.08.

b First extraction, 8.22; second, 0.31; third, 0.08.

c Modified method, 7.65.

TABLE V.—*Fat in sample No. 123, unsweetened condensed milk.*

Analyst.	Asbestos method.			Babcock test.		Gottlieb method.
	First extraction.	Second extraction.	Total.	Leach.	Straight.	
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
J. M. Barnhart, Illinois.....			8.02	8.4		
James M. Doran, Washington, D. C.....						7.96
J. A. Hummel, Minnesota.....					7.20	
Geo. A. Olson, Wisconsin.....	8.10	0.37	8.47	8.1	8.20	8.41
G. E. Patrick and M. Boyle, Washington, D. C.....	8.11	.01	a 8.12			b 8.49
P. H. Smith and E. B. Holland, Massachusetts.....	6.47	.74	c 7.21			
A. C. Whittier, Maine.....			7.91	8.1		8.32

* Adams paper coll. 40 per cent solution, first extraction, 8.11; second, 0.01.

b First extraction, 8.47; second, 0.02.

c Modified method, 7.40.

REMARKS BY ANALYSTS.

G. E. Patrick: For comparison, fat was determined by the Adams method on a 40 per cent solution of the sweetened condensed milk, giving a result—the mean of triplicates—about 0.3 per cent lower than by the same method on a 20 per cent solution, as given above.

In the Gottlieb method for fat the Rohrig tube, mentioned in last year's report, has been used continuously with the greatest satisfaction. We consider the Gottlieb method the best known for the determination of fat in milks.

Geo. A. Olson: Adhered to methods as described in circular letter.

P. H. Smith and Edw. B. Holland: Method (A). Extracted the pulverized solids with dry ethyl ether in a continuous extractor before and after washing to remove sugars. Method (B). Washed solids with water to remove sugars and extracted with ethyl ether. Results higher, with a considerable saving of time and work.

James M. Doran: One to one and a half grams of the original sample was used for the Gottlieb method and allowed to stand over night before siphoning. The Adams-Soxhlet method gave low results after three days' extraction. It appeared to be unsatisfactory owing to the length of time required to obtain results.

DISCUSSION OF RESULTS.

As stated last year, the double-extraction method increased the per cent of fat in both sweetened and unsweetened milk. The Gottlieb method generally gives higher results than any one of the other methods employed. The lack of agreement in the different methods of estimating the fat may be, in part, due to the variations in the samples.

SUGAR IN CONDENSED MILK.

TABLE VI.—*Sugar in sample No. 9, sweetened condensed milk.*

Analyst.	Method.	Gravimetric.		Polariscope.	
		Lactose.	Sucrose.	Lactose.	Sucrose.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
J. M. Barnhart, Illinois.....	Walker.....	^a 12.61			
A. H. Bryan, Washington, D. C.....	Soxhlet.....	^a 12.18	^a 42.49		
Do.....	Walker.....	^a 12.71			
Do.....	Soxhlet.....	^b 12.09	^b 42.56		
Do.....	Walker.....	^b 12.60			
Do.....	Soxhlet.....	^c 12.12	^c 42.50		
Do.....	Walker.....	^c 12.63			
Do.....	Soxhlet.....	^c 12.92	^a 42.56		
James M. Doran, Washington, D. C.....	Dubois.....	^a 12.68	^a 43.56		
A. Given, Washington, D. C.....	Walker.....	^a 12.96			
Do.....	Soxhlet.....	^b 12.37	^b 43.58		^d 42.20
Geo. A. Olson, Wisconsin.....	Allihn.....	^c 11.58	^c 43.14		43.51
G. E. Patrick and M. Boyle, Washington, D. C.....	Soxhlet.....				
Do.....	Walker.....	^c 11.62			
P. H. Smith and E. B. Holland, Massachusetts.....	Defren O'Sullivan.....	^c 11.97	40.81		
A. C. Whittier, Maine.....		12.65	42.75		41.30
M. H. Wiley, Washington, D. C.....	Allihn.....	^a 12.80	^a 43.67		

^a Weighed as C₁₂O.

^b Weighed as C₁₁O.

^c Low zinc acetate method.

^d In another sample 42.72 per cent sucrose was obtained.

^e Munson and Walker method.

TABLE VII.—*Sugar in sample No. 123, unsweetened milk.*

Analyst.	Method.	Gravimetric.		Polariscope.
		Lactose.	Sucrose.	Lactose.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
J. M. Barnhart, Illinois.....		12.17	None.	
A. H. Bryan, Washington, D. C.....	Walker.....	^a 11.38		
Do.....	Soxhlet.....	^a 11.51		
Do.....	Walker.....	^b 11.20		
Do.....	Soxhlet.....	^b 11.38		
Do.....	Walker.....	^c 11.35		
Do.....	Soxhlet.....	^c 11.50		
James M. Doran, Washington, D. C.....	Dubois.....	^a 11.86		
A. Given, Washington, D. C.....	Walker.....	^a 11.56		
Do.....	Soxhlet.....	^a 11.48		
George A. Olson, Wisconsin.....	Allihn.....	^b 10.90	None.	10.86
G. E. Patrick and M. Boyle, Washington, D. C.....	Soxhlet.....	^c 11.48		11.12
Do.....	Walker.....	^c 11.33		
P. H. Smith and E. B. Holland, Massachusetts.....	Defren O'Sullivan.....	^c 11.15	0.67	
A. C. Whittier, Maine.....		9.79	None.	
M. H. Wiley, Washington, D. C.....	Allihn.....	^a 11.56		

^a Weighed as Cu₂O.^b Weighed as CuO.^c Low zinc acetate method.

REMARKS BY ANALYSTS.

P. H. Smith and E. B. Holland: Inverted with one-tenth its volume of concentrated hydrochloric acid, neutralized with sodium hydrate solution and proceeded as under reducing sugar. Calculations after deducting for the lactose by association tables, pages 35 and 37, Bulletin 46, Revised edition. Dextrose to sucrose by factor 0.95.

James M. Doran: Trials with some of the well-known methods have proved unsatisfactory. With proper conditions, however, in the inversion and temperature it is thought that both sucrose and lactose can be determined accurately.

G. E. Patrick: Lactose corrected for error caused by cane sugar.

C. A. Browne: You will note a variation in the lactose in sample No. 9 by the different methods, which I attribute to the varying degree of inversion of sucrose produced by the different methods of procedure. The Walker method requires only two minutes instead of six as by the Soxhlet, and uses much less alkali than the Allihn with decidedly less inversion in each case. In sample No. 123, where no sucrose was present, the results by the three methods are in very close agreement.

The error due to the inversion of sucrose in the gravimetric determination of sugars in sweetened milks seems to be as pronounced as in the analysis of sugar cane or beet products. We had no time to determine what correction should be made for this, but the experiments performed upon mixtures of lactose and sucrose (see following tables) indicate that an error of at least 0.50 per cent is involved in the case of the determinations by the Allihn method. Weighing the cuprous oxid gave accurate results with all samples, there being no contamination of the precipitate with organic or with mineral matter, as is often the case with molasses.

Effect of inversion of sucrose on the determination of lactose (M. H. Wiley).

Amount taken.		Amount found.
Lactose.	Sucrose.	Lactose.
<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
2.00		1.97
2.00	10.00	2.75
4.00		3.97
4.00	10.00	4.72
8.00		8.03
8.00	10.00	8.92
12.00		12.17
12.00	10.00	12.84

The Allihn method was used in all determinations, the dextrose being converted to lactose by the formula $\frac{D}{0.675} = \text{lactose}$. The lactose used had one-half molecule of water of crystallization.

Analysis of an artificial mixture of sucrose and lactose as found in condensed milk.

[M. H. Wiley and C. A. Browne.]

Amount taken.		Amount found.		
Lactose (one-half molecule of water).	Sucrose.	Allihn's method.		Clerget's method.
		Lactose.	Sucrose.	Sucrose.
<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
12.00	42.00	12.60	41.47	42.14

Per cent sugar after inversion as dextrose equals 50.44; per cent sugar before inversion as dextrose equals 8.72; per cent reducing sugars as dextrose from sucrose equals 41.72.

Therefore $\frac{8.72}{0.692} = 12.60$ per cent lactose (one-half molecule of water).

$41.72 \times 0.993 = 41.47$ per cent sucrose.

DISCUSSION OF RESULTS.

The determination of sugars in condensed milk as reported this year indicates a very promising progress. The results as a whole show a fair agreement when one considers the variation in the total solids, which alone is sufficient to cause differences that would exceed those shown in the data reported. The determinations of sugars in unsweetened milk appear to be more uniform than those in sweetened milk. Polariscopic determinations for sucrose are generally lower than those obtained by gravimetric methods and may be attributed to the inversion of a part of the sucrose. The effect of inversion on the determination of lactose, brought about by the addition of sucrose, is interesting, and in consequence a higher lactose reading would be expected. It is evident that in future work along this line it will be necessary to determine the factor for correction in milk sweetened by sucrose.

RECOMMENDATION.

It is recommended—

(1) That the study of methods of analysis for condensed milk be continued and that special attention be given to the following methods for determining the fat and sugar content of condensed milk.

(a) Double extraction method for fat as described in the proceedings of the association for 1906.^a

(b) Gottlieb method as described in the proceedings of the association for 1906.^a

(c) Walker method for the gravimetric determination of sugar (J. Amer. Chem. Soc., 1907, 29: 541).

(d) Allihn method for the gravimetric determination of sugar.

(e) Polariscopic method for comparison with the gravimetric method for the determination of sugar.

G. E. PATRICK. In our laboratory we always correct the lactose result for the error caused by the presence of sucrose in analyzing sweetened condensed milks. We have not yet made a table of corrections, but have thus far determined the error in each sample by making a water solution of the two sugars corresponding to the results found, and in this solution—and a similar one of lactose without sucrose—finding the amount of the error in milligrams of copper oxid, which we then deduct from the copper oxid obtained in the analysis. In our experience thus far the correction has been from 12 to 15 milligrams by the Soxhlet method, and 8 to 12 milligrams by the Walker method on the charges prescribed by the official method for sweetened condensed milk.

P. H. WALKER. I have made a few determinations with mixtures of sucrose and lactose which would indicate that the correction needed would be very small. The lactose used, however, was the same as that employed in making up the tables published in the Journal of the American Chemical Society and had been standing for about eighteen months. Not sufficient work has been done to make up tables for this correction.

TWO METHODS IN CHEESE ANALYSIS.

By G. E. PATRICK.

The two following methods for the determination of moisture and the separation of fat in cheese analysis have been used with great satisfaction for several years in the Dairy Laboratory of the Bureau of Chemistry. They are presented to the association with the suggestion that it might be wise to include them in the methods, as in the writer's opinion they are far superior to the procedures there outlined.

1. WATER DETERMINATION.

Sift a quantity of asbestos by rubbing it through a wire screen—a window screen free from rust serves the purpose well. (Iron rust in the asbestos works mischief in the subsequent ash determination.) In a flat-bottomed platinum dish (aluminum may be used if the ash is not to be determined) of 6 to 7 cm diameter place about 2 grams (a little more rather than less) of the sifted

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 105, p. 105.

asbestos, pressing it down well over the bottom, then lay in the dish a thick glass rod cut off square at one end, to serve as a pestle. Ignite and weigh, then weigh into the dish about 5 grams of the cheese sample (prepared as usual) and with the glass rod rub and press the cheese and asbestos together most intimately, until all cheese particles have disappeared and the mass is homogeneous; finally, loosen the mass up into as fluffy a condition as possible, and place in the drying oven. Dry until loss of weight ceases, each drying to be for only an hour or an hour and a half. Usually two or three dryings suffice.

2. SEPARATION OF THE FAT FOR EXAMINATION.

Rub up thoroughly in a mortar 75 grams of the cheese with 90 to 100 cc of ether, throw the mass upon a cloth filter, press out the ether-fat solution as much as possible, return the mass to the mortar and repeat the treatment twice again (the second time less ether will suffice); filter the ether-fat solution through paper, recover the ether by distillation, filter the fat if it is not clear, then dry on the steam bath and finally in the oven until the ether is expelled, weighing every hour in order to guard against oxidation.

Petroleum ether boiling below 40° C. may be used, but has the fault of being expelled rather slowly from the fat. If only the neutral fat, free from uncombined fatty acids, is desired, add to the original extract two or three drops of phenolphthalein solution and then soda solution dropwise, with constant shaking, until the aqueous portion remains pink; then proceed as already described.

REPORT OF COMMITTEE C ON RECOMMENDATIONS OF REFEREES.

By L. M. TOLMAN, *Chairman*.

(1) MEAT PROTEIDS.

It is recommended that—

(1) The modified tannin-salt method for the separation of proteoses and peptones from the simpler amido bodies be adopted as a provisional method. (For statement of method, see referee's report, p. 51.)

The committee recommended that this method be further studied for another year before its adoption, and this motion was passed.

(2) That the following method for the determination of the acidity of meat products be adopted as a provisional method:

Determine the acidity by titrating a dilute solution of the meat, or meat preparation, with N/10 alkali, using phenolphthalein as indicator. But if the color prevents the use of this indicator, use litmus paper, with the understanding that the results are lower than when phenolphthalein is employed.

Adopted.

(3) That inasmuch as the best modification of the Folin colorimetric method for the determination of kreatin and kreatinin in meat products has not been determined, this point be studied another year.

Adopted.

(4) That the referee for 1908 investigate methods for the estimation of organic phosphorus and of xanthin bases.

Adopted.

(2) FLAVORING EXTRACTS.

It is recommended—

(1) That methods for the detection of caramel and other coloring matter in vanilla extracts be studied.

Adopted.

(2) That the quantity and quality of soluble matter in vanilla beans obtained in the preparation of standard vanilla extract be determined by the referee in 1908.

Adopted.

(3) DETERMINATION OF MOISTURE IN FOODS.

It is recommended that the work on this subject be continued another year.

Adopted.

(4) COLORS.

It is recommended that further work be done looking to a more comprehensive examination of the so-called "vegetable colors," some of which would seem to be no more properly classed as vegetable colors than are the anilin dyes.

Adopted.

(5) VEGETABLE PROTEIDS.

It is recommended that special attention be given to the study of methods for the determination of proteid and nonproteid nitrogen.

Adopted.

(6) COCOA AND COCOA PRODUCTS.

It is recommended that the methods submitted by the referee^a be adopted as provisional until a fuller report on the subject can be made, except that for section 13 on sugar the method proposed by Dubois for the determination of sugar in chocolate be substituted.

Adopted.

REPORT OF THE COMMITTEE ON THE REVISION OF METHODS.

J. K. HAYWOOD, *Chairman*.

The following report was presented by Mr. L. M. Tolman, acting for the chairman:

In accordance with the recommendations made in the report of the committee on revision of methods in 1906, and the suggestions of the secretary of the association made at that time, the revised methods were printed as Bulletin No. 107 of the Bureau of Chemistry and forwarded to the members of the association for criticism about three weeks before the 1907 meeting. Copies of this bulletin were sent to the following lists: Officers and referees of the association; directors and chemists of State agricultural experiment stations; superior officers of State boards of health, and chiefs of laboratories in the Bureau of Chemistry, Department of Agriculture.

The following letter was sent with each copy:

SEPTEMBER 12, 1907.

DEAR SIR: Inclosed you will find a copy of the revised methods of analysis of the Association of Official Agricultural Chemists recommended last year by the committee on revision and provisionally adopted by the association. Final action upon these revised methods will be taken this year.

You are respectfully requested to look these methods over at once, especially those with which you are most familiar. If you have any criticism to make of the changes incorporated, or can point out any errors, please report same to J. K. Haywood, Bureau of Chemistry, U. S. Department of Agriculture, Washington, D. C., chairman of revision committee, so that the letter will reach him before October 5, if possible, certainly not later than October 7. All criticisms will be considered by the committee at a meeting to be held in Washington on Octo-

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 107 Revised, p. 254.

ber 5 to 8, and recommendations made to the association at the Jamestown meeting on October 9 to 12, in regard to any further changes or additions, before the revised methods are officially adopted.

Respectfully,

H. W. WILEY,
Chief, Bureau of Chemistry,
Secretary, Association Official Agricultural Chemists.

Twenty-five chemists sent criticisms of the revised methods. These criticisms were carefully considered by this committee and, in the main, were adopted, the majority being simply corrections of obvious errors in spelling, punctuation, printing, etc. Some chemists suggested material changes in official methods, which were not adopted, as the committee did not feel that it had the power to make such far-reaching modifications. When changes were suggested to make the statement of the method more clear, but not to change the method itself, these were adopted.

The committee would respectfully recommend that the methods as now published in Bulletin 107 and further corrected, as indicated above, be adopted as the official and provisional methods of the Association of Official Agricultural Chemists.

(Signed)

J. K. HAYWOOD, *Chairman.*
 J. P. STREET,
 A. L. WINTON,
 F. P. VEITCH,
 L. M. TOLMAN,
 J. H. PETTIT,
 F. W. WOLL (absent).

Upon a motion being made to adopt the report, a discussion ensued as to the extent of the authority of the committee to change the methods by dropping obsolete procedures and otherwise bringing the methods up to date and the several sections into harmony with each other, especially when changes in official methods were involved.

The original motion was amended by a motion to continue the committee with authority to make such further changes of this nature as might be deemed necessary, said changes to be approved by the executive committee and inserted in the final revision. This motion was passed, and the original motion as amended was then carried, adopting the report of the committee and making the revision official.

A vote of thanks was passed by the association in appreciation of the work of the committee on revision.

REPORT ON FOODS AND FEEDING STUFFS.

By J. K. HAYWOOD, *Referee.*

On account of a great pressure of official work it was impossible for the referee to send out samples for examination; however, some comparative work was done by two chemists (Messrs. Goodrich and Houghton) of the Miscellaneous Laboratory of the Bureau of Chemistry, working entirely independently of each other.

The method of determining methyl pentosans, originated by Ellett and Tollens,^a and reported, in a modified form, by the referee last year, was first considered. Varying quantities of gum tragacanth, which is known to contain some methyl pentosans, were subjected to analysis by the following method:

METHOD FOR DETERMINING PENTOSANS AND METHYL PENTOSANS.

Proceed as in the determination of pentosans by the provisional method (Bulletin 107, page 54) until the phloroglucid precipitate has been dried for four hours and weighed. Place the Gooch crucible containing this precipitate in a 100 cc beaker and pour into the Gooch 30 cc of 95 per cent alcohol heated to 60° C. Place the beaker for ten minutes in a water bath heated to 60° C. Remove the beaker and Gooch and draw off from the Gooch with a suction pump all alcohol remaining therein. Repeat this alternate extraction and sucking dry of the precipitate from three to five times, according to the color of the filtrate obtained. After the final extraction place the Gooch crucible in a water oven and dry four hours, making the final weighing in a closely stoppered glass weighing bottle, as described in the provisional method for pentosans.

The difference in weight between the furfural-phloroglucid plus methyl-furfural-phloroglucid first obtained and the furfural-phloroglucid remaining after extraction with alcohol, minus 0.0037, represents the amount of methyl-furfural-phloroglucid present, from which the original pentose (as rhamnose) can be calculated by the following formula:

$$\text{Rhamnose} = (\text{Ph}) (165) - (\text{Ph})^2 (1.84) + 0.010.$$

in which "Ph" equals the weight of methyl-furfural-phloroglucid; rhamnosan equals rhamnose multiplied by 0.8. A table giving these calculated figures will be found on page 19 of the article by Ellett and Tollens,^a in which attention is directed to an error in the calculation of 0.028 and 0.029 gram of the methyl-furfural-phloroglucid to rhamnose.

To obtain the weight of pentosans, subtract the final corrected weight of methyl-furfural-phloroglucid from the weight of the mixture of methyl-furfural-phloroglucid and furfural-phloroglucid and calculate according to Kröber's table, or according to the formulas given in the present provisional methods for pentosans.

The following results were obtained by the two chemists engaged in the work:

TABLE I.—*Determination of pentosans and methyl-pentosans.*

GOODRICH'S RESULTS.

Gum tragacanth.	Furfural-phloroglucid + methyl-furfural-phloroglucid.	Loss by alcoholic extraction.	Pentosans.	Methyl-pentosans as rhamnosan.
Grams.	Grams.	Grams.	Per cent.	Per cent.
0.5	0.2258	0.0119	39.42	3.74
0.5	0.2256	0.0125	39.28	3.90
0.5	0.2295	0.0117	40.12	3.69
0.5	0.2185	0.0128	38.00	4.13
Average			39.21	3.87
0.8	0.3593	0.0167	38.52	3.11
0.8	0.3544	0.0178	38.11	3.27
0.8	0.3511	0.0170	37.83	3.16
0.8	0.3531	0.0182	37.92	3.35
Average			38.10	3.22

^a Jour. Landw., 1905, 53 (1): 13.

TABLE I.—*Determination of pentosans and methyl-pentosans—Continued.*

HOUGHTON'S RESULTS.

Gum tragacanth.	Furfural-phloroglucid + methyl-furfural-phloroglucid.	Loss by alcoholic extraction.	Pentosans.	Methyl-pentosans as rhamnosan.
Grams.	Grams.	Grams.	Per cent.	Per cent.
0.5	0.2294	0.0194	38.74	5.66
0.5	0.2266	0.0134	39.30	4.12
0.5	0.2320	0.0118	40.54	3.72
0.5	0.2318	0.0142	40.08	4.36
0.5	0.2228	0.0158	38.20	4.74
0.5	0.2346	0.0142	40.08	4.34
0.5	0.2338	0.0138	40.50	4.26
0.5	0.2328	0.0126	40.54	3.92
0.5	0.2358	0.0144	40.76	4.44
0.5	0.2426	0.0166	41.56	4.94
0.5	0.2372	0.0150	40.90	4.54
0.5	0.2356	0.0126	41.04	3.92
0.5	0.2354	0.0116	41.18	3.66
Average.....			40.26	4.36
0.8	0.3592	0.0196	38.43	3.58
0.8	0.3642	0.0216	38.88	3.89
0.8	0.3686	0.0210	39.32	3.79
0.8	0.3672	0.0220	39.10	3.96
0.8	0.3682	0.0182	39.18	3.35
0.8	0.3706	0.0182	39.85	3.35
0.8	0.3517	0.0160	38.01	3.00
0.8	0.3806	0.0236	40.34	4.21
0.8	0.3704	0.0186	39.79	3.41
0.8	0.3746	0.0174	40.35	3.22
0.8	0.3704	0.0158	40.09	2.96
Average.....			39.31	3.52

In interpreting these results, it must be borne in mind that gum tragacanth contains a very large amount of pentosans, larger, in fact, than almost any of the food or feed materials that the chemist will be called upon to examine; hence it was necessary to employ a small original weight of the gum, and this would result in small errors of weighing being very much magnified when the results were expressed on a percentage basis. Judging from Table I the method employed does not give as good results as was hoped. It will at once be seen from the work of both chemists that there is a tendency to get low results on methyl pentosans when the amount of gum tragacanth is increased. Expressing this in another way, there is a tendency for the amount of methyl pentosans to decrease as the weight of the furfural-phloroglucid + methyl-furfural-phloroglucid precipitate increases. This is probably due to the fact that the alcohol extraction is not so complete when a large precipitate is being handled. The results obtained by the same analyst on a definite weight of gum tragacanth do not vary from one another, in the large majority of cases, more than one would expect. The same can not be said of the results obtained by different analysts, which vary from each other more than is desirable even in such a conventional method. However, the averages obtained by the two analysts for methyl pentosans are close enough to warrant the referee for next year in sending out samples for further comparative work.

Even as the method stands it is worthy of adoption by the association as a roughly approximate method to be used by those who desire to make as complete an analysis as possible of vegetable materials. As the analysts become more familiar with the manipulation of the method, which is somewhat difficult and tedious, it is possible that their results will become more uniform. Better results will also be obtained upon the substances usually examined by feeding stuff methods than upon gum tragacanth, in which the amount of furfural-phloroglucid is so large that the extraction of the methyl-furfural-phloroglucid

is somewhat incomplete if a moderate quantity of the original gum is used. I would therefore recommend that the method be further studied next year, especial attention being given to obtaining a large amount of comparative work.

Since calculating the percentages of methyl pentosans (as rhamnosan) from the formula given by Ellett and Tollens, an article by Mayer and Tollens^a has appeared in which is given the formula for calculating the methyl-pentosans to fukose and also a table for calculating methyl-furfural-phloroglucid to fukose, fukosan, rhamnose, rhamnosan, and methyl pentosan (average of fukosan and rhamnosan).

I would, therefore, further recommend that this table be used in calculating the results of analysis, the average figure for methyl pentosans being taken when it is not known whether the mother product is fukosan or rhamnosan.

In accordance with instructions from the association, a study was also made of the time required to dry the crude fat obtained in the ether extract determination on several different feeding stuffs. The dryings were made at the temperature of boiling water and the following feeding stuffs were employed: Wheat germ meal, ground corn, cotton-seed meal, and flaxseed. To be certain that all fat was extracted from the samples, the time of extraction with ether was increased from sixteen to about twenty-four hours. The following results were obtained by the two chemists engaged in the work:

TABLE II.—*Determination of time required to dry the crude fat obtained in ether extracts from different feeding stuffs.*

GOODRICH'S RESULTS.

Material.	Weight of sample.	Crude fat extracted.					
		½ hour.	1 hour.	1½ hours.	2 hours.	2½ hours.	3 hours.
	Grams.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Wheat germ meal.....	1	5.76	4.24	3.89	3.77	3.73	3.73
Do.....	2	4.30	4.00	3.94	3.90	3.88	3.89
Do.....	3	4.03	3.95	3.94	3.92	3.91	3.92
Do.....	4	3.82	3.80	3.86	3.86	3.88	3.89
Do.....	4	3.80	3.76	3.77	3.78	3.80	3.81
Do.....	4	3.77	3.72	3.79	3.80	3.81	3.82
Do.....	4	3.87	3.84	3.89	3.86	3.93	3.92
Do.....	4	3.88	3.85	3.89	3.86	3.93	3.92
Do.....	4	3.86	3.83	3.89	3.86	3.92	3.90
Corn.....	1	4.95	4.76	4.72	4.64	4.69	4.69
Do.....	2	5.69	4.80	4.67	4.61	4.61	4.59
Do.....	3	5.08	4.60	4.49	4.46	4.47	4.47
Do.....	4	4.17	4.16	4.19	4.22	4.23	4.23
Do.....	4	4.22	4.14	4.16	4.17	4.23	4.24
Do.....	4	4.36	4.21	4.22	4.23	4.28	4.31
Cotton-seed meal.....	2	11.44	11.37	11.30	11.33	11.33	
Do.....	2	11.45	11.40	11.29	11.35	11.36	
Do.....	2	11.70	11.50	11.36	11.41	11.43	
Linseed.....	2	27.70	26.85	26.64	26.98	27.02	
Do.....	2	27.38	26.54	26.43	26.46	26.45	

HOUGHTON'S RESULTS.

Wheat germ meal.....	1	4.28	3.92	3.92	4.00	4.02	3.98
Do.....	2	4.45	3.74	3.68	3.65	3.70	3.77
Do.....	3	4.01	3.84	3.83	3.86	3.88	3.92
Do.....	3	4.71	3.91	3.87	3.83	3.85	3.89
Corn.....	1	4.72	4.58	4.80	4.73	4.62	4.62
Do.....	1	6.46	4.36	4.28	4.44	4.42	4.62
Do.....	2	5.19	4.48	4.36	4.44	4.55	4.52
Do.....	3	4.56	4.27	4.23	4.27	4.23	4.25
Do.....	3	4.89	4.33	4.30	4.36	4.36	4.35
Cotton-seed meal.....	1	11.56	11.22	11.22	11.26	11.26	11.34
Do.....	1	12.40	11.24	11.12	11.00	11.06	11.34
Linseed.....	1	27.24	26.62	26.48	26.52	26.54	26.70
Do.....	1	28.60	26.88	27.02	26.92	26.92	26.98
Do.....	1	28.46	26.84	26.64	27.02	27.36	27.24
Do.....	1	27.56	26.38	26.56	26.94	27.18	27.08

^aJour. Landw., July, 55: 269.

It is at once evident from these results that no definite period for drying the crude fat can be specified in the method for determining this substance in cattle food materials. The time necessary to obtain a minimum weight evidently varies with the individual performing the work, with the amount of substance taken, with the character of the fat extracted, with the amount of fat in the substance, and possibly with other factors. A second point brought out is one that would be expected, i. e., that there is a marked tendency for the fat to reach a minimum weight and then slowly to gain by oxidation of the fat. This is, of course, particularly well marked in the case of linseed, which contains an oil that has a high oxygen-absorbing power.

A third point made evident by this investigation is, that in every case at least a one-hour period of drying was necessary to reach a minimum weight, while in some cases this period was extended to two and one-half hours. In the large majority of cases a one and one-half hour period of drying the crude fat was sufficient to obtain either a minimum or results so close to the minimum that the difference was negligible. On the whole, then, while a definite time of drying the fat can not be specified, it may be of some service to the inexperienced to state that the time is usually between one and one-half hours.

I would recommend, therefore, that the last sentence under Crude Fat, or Ether Extract, Direct Method, page 39, Bulletin 107 (Methods of Analysis), which now reads, "Dry the extract at the temperature of boiling water until it ceases to lose weight," be changed to read, "Dry the extract at the temperature of boiling water for one-half hour, remove from the oven to a desiccator, let it cool, and weigh; continue this every alternate one-half hour, drying and weighing until a minimum weight of the fat is obtained. For most substances a period of one to one and one-half hours is required to obtain a minimum weight."

REPORT ON SPECIAL ANALYTICAL METHODS OF SUGAR ANALYSIS.

By C. A. BROWNE, *Referee*.

The work of the referee on special methods of sugar analysis for the present year has covered the investigation of a number of carbohydrate products, but the present report will be limited to a discussion of only three of these: First, the analysis or assay of commercial dextrans; second, a comparison of methods for the estimation of glucose in honey, and third, a study of methods for detecting artificial invert sugar in honey.

ASSAY OF COMMERCIAL DEXTRANS.

During the past year the Bureau of Chemistry has had occasion to analyze a number of dextrans for the work of the Bureau of Engraving and Printing. As a basis for methods an extensive article by Friedrich Lippmann^a upon the "Examination and analysis of commercial dextrans" was used. An abstracted translation of this article has been prepared by the referee, and copies so far as available can be furnished to members of the association.

The methods described in this abstract for moisture, ash, insoluble organic matter, and reducing sugars, were followed, and the difference between these and 100 per cent taken as dextrin, as advocated by Lippmann. It should be noted, however, that this difference includes other cold-water soluble organic materials besides dextrin, such as acids, proteid bodies, and various decomposition products of a carbohydrate nature formed during the process of dextrin-

^a Zts. Spiritus-Ind., 25: 304, 307, 316, 317.

ization. These bodies should not, of course, be included with the dextrin, yet from the fact that there is no satisfactory method for the quantitative isolation of the dextrin from the accompanying impurities, the dextrin is taken as difference. For the specific purpose for which the dextrins are used at the Bureau of Engraving and Printing it was necessary to make a somewhat closer differentiation of the cold-water soluble matter (even if it were only an arbitrary one) which would throw more light upon the character of the products. Therefore the specific rotation of the product was first determined; the specific rotation equivalent to the reducing sugars present as dextrose was then subtracted from this and the remainder taken as the specific rotation due to the pure dextrin present. This remainder multiplied by 100 and divided by 186, the specific rotation of pure dextrin,^a will give the percentage of dextrin present. The difference between the sum of the constituents by this method of calculation and 100 is taken as the undetermined solubles. While the polariscopic determination of dextrin, by such a method as the one described, is of but little scientific value for the reasons given by Lippmann, it is believed that the results obtained by it have a decidedly practical value and give the chemist a more accurate idea of the character of his product than where dextrin is estimated simply by difference.

In the following table results obtained by the method described upon eleven samples of commercial dextrin are given. It will be noted that the results for dextrin obtained from specific rotation are more nearly in accord with the relationship indicated by viscosity than by the estimation of dextrin by difference.

Analysis of dextrins.

Number.	Specific rotation [α] _D	Viscosity (1:2 solution water = 100).	Moisture 105° C.	Ash.	Reducing sugars as dextrose.	Cold water, insoluble, organic.	Dextrin by difference.	Dextrin from polarization.	Undetermined soluble.	Acidity N ₁₀ NaOH per 10 grams).
			Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	cc.
1	+157.7	250	6.23	0.07	1.80	0.32	91.58	84.34	7.24	2.0
2	+173.9	425	1.59	.12	2.16	.23	95.90	92.94	2.96	2.4
3	+163.1	275	4.69	.17	4.26	.55	90.33	86.53	3.80	2.4
4	+166.9	338	2.48	.06	3.34	.18	93.94	90.47	3.47	3.5
5	+170.6	450	4.13	.07	2.16	.51	93.13	91.18	1.95	2.5
6	+166.4	450	4.70	.10	1.43	.67	93.10	90.52	2.58	2.5
7	+148.3	200	3.98	.10	6.77	1.25	87.90	77.87	10.03	2.4
8	+171.5	368	2.59	.18	2.15	.44	94.63	91.69	2.94	2.6
9	+157.9	263	3.11	.16	4.59	.72	91.42	83.65	7.77	2.8
10	+163.1	313	5.21	.09	3.05	.44	91.21	86.88	4.33	2.4
11	+162.9	350	7.43	.06	1.85	.98	89.68	87.13	2.55	2.3

The viscosity determination is of paramount value as a physical test, the results by this, corresponding most closely with the practical results to which the dextrin is put. Arranging the samples in order of viscosity, it will be seen that the value of a dextrin, as would be expected, is inversely proportional to the amount of undetermined solubles.

The following are offered to the association as provisional methods for the analysis of commercial dextrins:

METHODS OF ANALYSIS.

Physical constants.

Specific rotation.—Transfer 10 grams of the finely divided sample to a 100 cc flask, and after solution in about 50 cc of cold water add 5 cc of alumina cream and make up the contents to 100 cc, thoroughly shake, and filter. Polarize the

^a L. Schulze, J. prakt. Chem., 1883, 28: 327.

filtrate in a 200 mm tube, using an S. and H. type of saccharimeter. Specific rotation $(\alpha)_D = \frac{V \ 34.68}{20}$, in which V=Ventzke reading.

Viscosity.—Dissolve 100 grams of dextrin in 200 cc of cold water and determine the viscosity of the solution by any of the standard forms of viscosimeter. Comparative results should always be made by the same instrument and under similar conditions of temperature. Results are expressed upon the basis that water equals 100.

Chemical analysis.

Moisture.—Determine by drying from 2 to 5 grams of sample for four hours at a temperature of 105° C. Absolute constancy in weight can not be attained on account of the slow decomposition of the dextrin.

Ash.—Determine by the official method of the association, Bulletin 107, Bureau of Chemistry.

Soluble starch.—If a filtered hot-water solution of the dextrin gives a strong blue reaction with iodine, soluble starch is indicated. Weigh two lots of dextrin, 10 grams each, into a 100 cc flask, add 50 cc of cold water to each, and after all soluble matter is dissolved make up the contents of the one flask with cold water to 100 cc, shake, and filter. Evaporate 20 cc of the solution (2 grams) to dryness and dry for four hours at 105°, as under determination of moisture. Weight of residue less ash on incineration equals cold-water soluble organic matter. Heat the contents of the second flask to boiling, and then after cooling make up to 100 cc, shake, and filter. The weight of hot-water soluble organic matter in 20 cc of solution is determined as before. Hot-water soluble organic less cold-water soluble organic gives the soluble starch.

Unconverted starch.—If the residue insoluble in hot water shows under the microscope grains which are colored blue with iodine, unconverted starch is present. To determine the percentage, collect the residue insoluble in hot water on a filter, wash until free from soluble matter, and determine the starch by the usual method.

Reducing sugars.—Determine in an aliquot of the cold-water soluble by the method of Allihn, the results being expressed as dextrose.

Dextrin.—Subtract the specific rotation of the dextrin due to reducing sugars $\left(\frac{52.5 \times \text{per cent reducing sugar as dextrin}}{100} \right)$ from the original specific rotation of the sample. Multiply the remainder by 100 and divide by 186 ($[\alpha]_D$ for pure dextrin) to obtain the calculated percentage of dextrin in the sample.

Undetermined solubles.—The per cent of cold-water soluble organic matter less calculated percentage of dextrin gives the percentage of undetermined solubles.

The other two studies on a comparison of the methods for the estimation of glucose in honey and the detection of invert sugar in honey are reported in full in Bulletin 110 of the Bureau of Chemistry, entitled, Chemical Analysis and Composition of American Honeys; they are, therefore, omitted from the Proceedings.

REPORT ON SUGAR AND MOLASSES METHODS.

By C. A. BROWNE, *Referee*, and J. E. HALLIGAN, *Associate Referee*.

The samples sent out for cooperative work by the referees on sugar and molasses consisted of a sample of Louisiana low-grade sugar and one of molasses obtained from Dr. G. L. Spencer, Pijuan, Cuba, representing the final

product of a Cuban sugar house. The following circular letter of instructions was sent to the cooperating chemists:

INSTRUCTIONS FOR ANALYSIS OF SUGAR AND MOLASSES SAMPLES, 1907.

I.—Moisture.

The objections raised at the last meeting to the method of drying ten consecutive hours (Bulletin 46, p. 27), thus exceeding the length of the usual laboratory day, renders further work upon the determination of water in molasses necessary. It is recommended that the method of Pellet (International Sugar Journal, 1906, 8: 500) be tested for this purpose. The method reads as follows:

Weigh 3 grams of molasses into a flat-bottom dish containing about 10 grams of finely crushed pumice stone and note the total weight. Dilute the molasses with a little boiling water, add one drop of ammonia and mix. Dry at 102° to 105° C. to constant weight, which will be complete in from two and one-half to three and one-half hours if the drying oven be previously heated to the required temperature.

Compare Pellet's method with the method of drying in vacuo, with any of the official methods, or with any other method preferred by the analyst.

Determine moisture in the sugar by the ordinary methods.

II. Determination of reducing sugars in molasses and sugar.

A. Clarify the solutions with lead subacetate in the usual way, removing any excess of lead with sodium carbonate (Bulletin 46, p. 33) or potassium oxalate (Circular 26, page 5).

(1) Determine reducing sugars by the method of Allihn (Bulletin 46, p. 35) and determine the reduced copper:

(a) From weight of Cu_2O .

(b) From weight of CuO (oxidation of Cu_2O after weighing by heating in a muffle).

(c) Volumetrically by Low's method.

The copper from (b) is dissolved in nitric acid and the copper determined either by Low's modified bromin method (Proceedings of 1905, Bulletin 39, p. 19) or the original Low zinc-acetate method. (J. Amer. Chem. Soc., 1902, 24: 1082.)

(2) Determine reducing sugars by method of Munson and Walker. (J. Amer. Chem. Soc., 1906, 28: 663.)

B. Make the same determinations on the filtered solutions without clarification with lead subacetate.

III. Polarization, direct and invert, of molasses and sugar.

Weigh out a normal weight and make to 100 cc or to such multiple thereof as may be necessary to secure an accurate polarization after clarifying as follows:

(a) With lead subacetate solution (Bulletin 46, pp. 38-39).

(b) With Horne's dry lead subacetate (J. Amer. Chem. Soc., 1904, 26: 186).

(c) With alumina cream and a little sodium hydrosulphite (R. A. S. F. preparation the best).

If there is time, compare the sucrose determination by double polarization with the gravimetric method.

Record carefully all temperatures of polarization, dilutions, etc., that results may be compared on as uniform a basis as possible.

It is also urged that the work on the samples be begun if possible immediately upon their arrival to avoid any changes in composition which might result from fermentation.

C. A. BROWNE, Jr., *Referee on Sugar.*

J. E. HALLIGAN, *Associate Referee on Molasses Methods.*

SUGAR.

The cooperative work on sugar for the year consisted in a comparison of different methods of clarification; first, as to their effect on the direct and invert polarization; and second, as to their effect on the determination of reducing sugars.

The results of the different chemists on the polarizations of sugars are given in the following table:

TABLE I.—*Polarization of sugar with different methods of clarification.*

[Normal weight to 100 cc, 200 mm tube.]

Clarifying agent and analyst.	Amount of clarifying agent.	Direct polarization.	Corrected invert polarization.	Temperature of polarization.	Sucrose by Clerget's method (factor 142.66).
Subacetate of lead:	cc.	° V.	° V.	° C.	Per cent.
A. W. Blair, Florida.....	5	82.6	—26.8	31	86.03
C. A. Browne, Washington, D. C.....	5	82.8	—29.6	26	86.68
A. H. Bryan, Washington, D. C.....	6	82.8	—32.0	20	86.53
A. Given, Washington, D. C.....	6	83.0	—30.0	20	85.18
J. E. Halligan, Louisiana.....	7	82.5	—26.6	29	85.13
W. D. Horne, New York a.....	6	82.0	—28.8	24.5	84.96
W. P. Naquin, Louisiana.....	5	83.1	—28.1	31	87.45
M. H. Wiley, Washington, D. C.....		82.7	—27.7	29	86.14
Fritz Zerban, Louisiana.....	5	83.2	—30.6	25	87.43
Average.....		82.84			86.32
Dry subacetate of lead:	Grams.				
C. A. Browne, Washington, D. C.....	1.0	82.4	—29.3	26	86.12
A. H. Bryan, Washington, D. C.....	2.0	82.8	—32.7	20	87.02
A. Given, Washington, D. C.....	2.0	82.7	—28.0	20	85.45
J. E. Halligan, Louisiana.....	1.5	82.5	—26.4	30	85.30
W. D. Horne, New York a.....	0.8	81.6	—27.6	24	83.62
W. P. Naquin, Louisiana.....		82.6	(b)		
M. H. Wiley, Washington, D. C.....	1.0	82.6	—28.2	28.5	86.52
Fritz Zerban, Louisiana.....		83.0	—30.8	25	87.43
Average.....		82.66			85.97
Neutral lead acetate:	cc.				
C. A. Browne, Washington, D. C.....	2.5	82.1	—29.2	26	85.80
A. H. Bryan, Washington, D. C.....	5	82.4	—32.2	20	86.39
J. E. Halligan, Louisiana.....		82.0	—25.6	30	84.29
M. H. Wiley, Washington, D. C.....	5	82.5	—28.8	29	86.81
Average.....		82.25			85.82
Alumina cream and sodium hydrosulphite:	Grams.				
C. A. Browne, Washington, D. C.....	0.1	81.9	—29.4	26	85.82
A. H. Bryan, Washington, D. C.....	0.5	81.4	—32.9	20	86.16
A. Given, Washington, D. C.....	0.3	82.5	—30.2	20	84.96
J. E. Halligan, Louisiana.....	0.5	81.7	—26.8	30	85.00
W. P. Naquin, Louisiana.....		82.8	—27.0	32	86.69
M. H. Wiley, Washington, D. C.....		82.4	(b)		
Fritz Zerban, Louisiana.....		82.8	(c)		
Average.....		82.21			85.73

^a Results of W. D. Horne omitted from average; the sugar was not analysed immediately on its arrival and had evidently deteriorated through fermentation.

^b Too dark to read.

^c Precipitation of sulphur prevented readings.

As regards direct polarization, the results of the different chemists, considering the variations of temperature, etc., under which the work was performed, show a fairly uniform agreement with each method of clarification. The percentages of sucrose by the Clerget method, however, show extremely wide variations. These are due, no doubt, partly to differences in the method of inversion and also to variation in dilution of the inverted solution, which was always darker. There is probably no determination where the element of personal error or equation is more evident than in the Clerget method for sucrose. This fact and the numerous inherent errors in the method has led Koydl^a, in a recent article, to recommend the complete abandonment of the Clerget process in analytical work and the adoption of the gravimetric process.

Comparing the direct polarizations, which have the greatest interest from a commercial point of view, a number of interesting facts are noted in connection

with the different methods of clarification. But before taking these up it may be well to refer briefly to several of the factors which have a bearing on the subject. The true polarization of a raw sugar is represented by the sum of the polarizations of the various optical constituents present, which include not only sucrose, but also the reducing sugars, dextrose, levulose, and other products, such as caramel, amids, amino acids, etc. The differences obtained by the use of different clarifying agents are due partly to the selective action of the precipitant upon the optical bodies other than sucrose and also partly to the volume of the precipitate. In the table it is seen that the average of the results when the subacetate of lead solution is used is 0.15 per cent higher than that obtained with the dry subacetate. This difference, while not noticeable in the work of each analyst, is nevertheless well marked and is to be explained by the greater concentration of the solution by the volume of the lead precipitate. Comparing these solutions with the one clarified with neutral lead acetate, we note that the latter gives a polarization decidedly lower, notwithstanding the volume of precipitate error. This is to be explained by the fact that subacetate of lead, whether wet or dry, is a powerful precipitant of the reducing sugar levulose, thus increasing the dextrorotation. Neutral acetate of lead has little if any action upon the reducing sugars.

The lowest readings of all were obtained with alumina cream and hydrosulphite of soda, and these readings ought to represent most closely the true polarization of the sugar, since there is no volume of precipitate error and no precipitation of reducing sugars. Hydrosulphite, however, if allowed to act for any length of time, seems to lower the polarization of some of the component sugars, though just how this is brought about is uncertain. With sucrose it seems to be due to a slight inversion, with dextrose it may result from the formation of sulpho-compounds of lower specific rotation. The referee has deferred the addition of the hydrosulphite until just before polarizing, adding a few small crystals to the solution in the tube before making the reading. An instantaneous decolorization is produced, and a reading can be taken before the sugars are appreciably acted upon. This can be done even in the inverted solution, the liquid remaining clear for five minutes before turbidity begins through decomposition of the hydrosulphite.

The effect of different clarifying agents upon mixed solutions of sucrose, dextrose, and levulose, in the presence of inorganic and organic matter precipitable by lead has been studied at the Bureau of Chemistry by Mr. A. H. Bryan. The work is not yet completed but the results of one set of experiments are given in the following table:

TABLE II.—*Polarization of mixtures of sucrose, dextrose, and levulose with 0.5 gram ammonium oxalate and 0.5 gram sodium sulphate using different clarifying agents.*

No.	Amount of clarifying agent.	Clarifying agent.	Direct polarization.
	cc.		Per cent.
1	5	Alumina cream.....	89.00
2	3.5	Lead subacetate solution.....	89.50
3	7	do.....	89.55
4	3	Normal lead acetate solution.....	89.20
5	6	do.....	89.20
6	1.5	Dry lead subacetate.....	89.05
7	4	Alkaline lead nitrate solution.....	89.00
8	1	Sodium hydrosulphite.....	88.60

g Grams.

Taking the experiment with alumina cream as the true polarization, it is seen that the lead subacetate solution gives a reading 0.5 per cent too high, and the normal acetate 0.2 per cent too high. The excess reading in the second experiment is due to volume of precipitate and in the first to both volume of precipitate and precipitation of levulose. The dry lead and alkaline lead nitrate give readings in this particular experiment exactly identical with the true polarization. This might seem at first sight to indicate no precipitation of optically active bodies; such a precipitation does take place, however, as will be shown later, and the experiment only shows that in this particular instance the dextrose and levulose were precipitated together in quantities sufficient to neutralize one another.

The determination of reducing sugars in the sugar by the method of Allihn and of Munson and Walker are given in the following table:

TABLE III.—*Determination of reducing sugar in sugar as dextrose by Allihn's and by Munson and Walker's method, using different clarifying agents.*

Clarifying agent and analyst.	Strength of solution.	Precipitant.	Weighing as Cu_2O .		Weighing as CuO .		Titration of copper. (Low's method.)	
			Allihn method.	M. and W. method.	Allihn method.	M. and W. method.	Allihn method.	M. and W. method.
None:			<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
A. H. Bryan.....	26 gm. to 100.		6.45	6.29	6.22	5.98	5.88	5.83
J. E. Halligan.....	1 gm. to 100.		6.94	6.43	7.05	6.51	7.02	6.37
W. D. Horne ^a	4 gm. to 100.		7.08	6.43	7.05	6.51	7.02	6.37
Average.....			6.82	6.36	6.63	6.25	6.45	6.10
Subacetate lead:								
A. W. Blair.....	{ 10 gm. to 200. 100 cc to 200.	{ 2½ cc.	6.33	5.77				
A. H. Bryan.....	{ 26 gm. to 100. 10 cc to 100.	{ 6 cc.	6.14	5.76	5.67	5.51	5.67	5.30
J. E. Halligan.....	{ 1 gm. to 100. 1 gm. to 100.	{ 2 cc. 4 cc.	6.65 6.44					
W. D. Horne ^a	{ 26 gm. to 100. 25 cc to 100.	{ 6 cc.	6.61	6.19	6.51	6.01	6.51	5.99
Average.....			6.43	5.91				
Dry subacetate lead:								
A. H. Bryan.....	{ 26 gm. to 100. 10 cc to 100.	{ 3 gr.	6.05	5.75	6.00	5.72	6.02	5.62
J. E. Halligan.....	{ 1 gm. to 100. 1 gm. to 100.	{ ½ gr.	6.67					
Average.....			6.36					
Neutral lead:								
J. E. Halligan.....	1 gm. to 100.	2 cc.	6.92					

^a The high determinations of Mr. Horne are probably due to a slight deterioration of the sugar through fermentation.

The highest results are obtained when no clarifying agent is used or only neutral acetate of lead; the results after clarifying with subacetate of lead solution or dry subacetate of lead are about 0.5 lower. The French chemists, and more lately the English chemists, have declared against the use of basic lead acetate in clarification for the determination of reducing sugars, and their contention seems wholly justified. To determine the extent of this error in analytical work the following series of experiments were carried out at the Bureau of Chemistry by Mr. A. H. Bryan. Solutions of dextrose and levulose were prepared, using 5 grams of sugar and 1 gram each of magnesium sulphate and ammonium tartrate. To 50 cc of this solution the precipitant was added and the volume made up to 100 cc. After filtering the excess of lead was removed with potassium oxalate and the sugar determined by Allihn's method.

TABLE IV.—Percentages of dextrose and levulose removed by different precipitating agents (A. H. Bryan).

[1 gram of magnesium sulphate and 1 gram of ammonium tartrate added to each 5 grams of sugar in solution.]

Precipitating agent.	Amount per 100 cc of solution.	Dextrose removed.	Levulose removed.
Normal lead acetate solution.....	cc.	Per cent.	Per cent.
Do.....	3.5	0.93	0.00
Lead subacetate solution.....	7.0	0.84	0.00
Do.....	3.5	3.35	8.03
Dry lead subacetate.....	7.0	8.34	19.91
Do.....	1.0	3.85	14.93
Alkaline lead nitrate (Herle's solution).....	2.5	17.48	35.33
Do.....	4.0	6.27	13.84
Do.....	8.0	5.61	25.12

a Grams.

The normal acetate of lead removes practically no reducing sugar in any of the experiments; the lead subacetate, on the other hand, in any of its forms, removes very large quantities of both dextrose and levulose, the latter sugar always in much larger amounts. In our present official methods for reducing sugars in saccharine products, wines, fruits, etc., the subacetate of lead is always prescribed for clarification. In view of the manifest error involved in the use of this reagent, it is recommended that its use be discontinued by the association in the determination of reducing sugars.

The results obtained by the different methods of estimating copper show, as was indicated last year, the highest result by the method of weighing as cuprous oxid (Cu_2O), and the lowest result by the Low method of titration. This is due to the contamination of the cuprous oxid with slight amounts of organic and mineral matter. With products of very low purity it is always safer to make a direct determination of the copper than to weigh the precipitated oxid.

MOLASSES.

DETERMINATION OF TOTAL SOLIDS.

The comparison of methods upon total solids made last year was again continued, the object of the cooperative work being to find a simple and rapid method which would give results agreeing closely with the method of drying in vacuo.

The results of the work are given in the following table:

TABLE V.—Determination of total solids in molasses.

Analyst	1 gram at 98°.			2 grams, at 98°.	2 grams, at 103°.	Pellet method, 103°.		Vacuum.	
	2 hours.	3 hours.	4 hours.	10 hours.	6 hours.	3½ hours.	5 hours.	98°, 7½ hours.	To constant weight.
A. H. Bryan, Washington.....	P. d.	P. d.	P. d.	P. d.	P. d.	P. d.	P. d.	P. d.	P. d.
J. E. Halligan, Louisiana.....				75.39	74.98	77.60	74.21		76.87
W. D. Horne, New York.....			75.47			74.64		75.54	
M. M. Klaverweiden, Cuba.....				74.89					
W. P. Naquin, Louisiana.....				76.76					
G. L. Spencer, Cuba.....	76.29	76.24							
A. Wedderburn, Cuba.....									76.70
F. Zerban, Louisiana.....				76.36					
Average.....	76.29	76.24	75.47	75.79	74.93	76.12	74.21	75.54	76.79

a 38 hours at 70° C.

In commenting upon the above, W. D. Horne remarks that the Pellet method of heating to 105° gives too high results owing to caramelization.

J. E. Halligan, in his method of drying six hours at 104° in a salt-water bath, obtained results agreeing closely with the method of drying 2 grams ten hours at 98°.

Mr. Bryan and Mr. Wedderburn by the method of drying in vacuum to constant weight obtained closely agreeing results, and the figures by this method, according to Mr. Spencer, are in best concordance with the workings of the sugarhouse.

The polarizations of the molasses by the different chemists using various methods of clarification are given in the following table:

TABLE VI.—Polarization of molasses with different methods of clarification.

Clarifying agent and analyst.	Half normal weight to—	Amount of clarify- ing agent.	Polarization.				Sucrose by Clerget method (factor 142.66).
			Direct.		Invert.		
Subacetate of lead filtered and acetic acid added until neutral or slightly acid:	cc.	cc.	° V.	° C.	° V.	° C.	° V.
J. E. Halligan, Louisiana	100	11.0	25.6	30.0	—20.0	30.0	35.72
W. D. Horne, New York	200	50.0	25.2				
M. Klaverweiden, Cuba	100	20.0	26.6				
W. P. Naquin, Louisiana	200	9.5	26.0	30.0	—17.6	31.0	34.29
G. L. Spencer, Cuba	100	20.0	26.6		—19.0	23.5	34.85
Fritz Zerban, Louisiana	200	9.5	27.2	28.5	—16.8	28.0	34.19
Average.....			26.2				34.76
Subacetate of lead polarized without acetic acid:							
A. H. Bryan, Washington	200	8.0	24.4	20.0	—24.6	20.0	36.95
W. D. Horne, New York	200	50.0	26.0	26.0	—18.4	26.0	34.24
M. Klaverweiden, Cuba	100	20.0	28.6				
G. L. Spencer, Cuba	100	20.0	28.8		—19.2	23.7	36.69
Average.....			26.95				35.96
Dry subacetate of lead:		Grams.					
A. H. Bryan, Washington	100	3.0	25.0	20.0	—20.7	20.0	34.45
J. E. Halligan, Louisiana	100	6.0	28.3	30.0	—17.5	30.0	35.87
W. D. Horne, New York	200	6.74	26.2	22.0	—19.2	22.0	34.48
W. P. Naquin, Louisiana	200		25.6	30.0	—18.4	30.0	34.46
Fritz Zerban, Louisiana	200		(c)				
Average.....			26.27				34.81
Hydrosulphite and alumina cream:							
A. H. Bryan, Washington	200	3.0	18.8	20.0	—26.4	20.0	34.07
J. E. Halligan, Louisiana	200	.5	23.8	30.0	—18.9	30.0	33.44
W. D. Horne, New York			(c)				
W. P. Naquin, Louisiana			(c)				
Fritz Zerban, Louisiana	200		27.2	29.0			
Average.....			23.27				33.76
Hydrosulphite added after subacetate of lead:							
A. H. Bryan, Washington	100		20.6	20.0	—24.2	20.0	33.77
M. Klaverweiden, Cuba	100		25.6		(c)		
G. L. Spencer, Cuba	100		27.0		(c)		
Average.....			24.4				33.77

^a 24 Brix lead subacetate.

^b Lead not removed prior to inversion (Prinsen-Geerligns method).

^c Too dark to read.

In the factory method employed by G. A. Spencer in Cuba, one-half the normal weight is made up to 100 cc after clarifying with lead subacetate solution (54.3° Brix), using as little as will give a suitable solution; 50 cc of the filtered

solution are acidulated with dilute acetic acid to decompose the soluble levulose of lead, and the volume is completed to 55 cc. The polariscopic reading, increased by one-tenth, is multiplied by 2 to give the corrected direct reading. Doctor Spencer states that the direct polarization is always too high when the excess of lead is not removed or neutralized with acetic acid.

In the use of potassium oxalate for removing lead, a number of the analysts experienced the difficulty of turbid filtration. W. D. Horne states that this difficulty can be obviated by the use of a little alumina cream.

The results confirm the observations made on the polarization of the raw sugars, namely, that higher results were secured with the wet subacetate of lead than with the dry and that the lowest results were obtained with the hydrosulphite. The question of the influence of the several clarifying and decolorizing agents has already been discussed under sugar and need not be referred to again here. The variations in the direct polarization of the molasses in each series of experiments show in many instances exceedingly wide variations, a circumstance due largely to difference in the temperature of polarization. These differences, it will be noted, are largely equalized in the calculations of the sugar by the Clerget formula, where the variations between the different chemists are less pronounced. The reverse of this was the case in the analysis of the sugar. The exceedingly large dilution necessary to secure a clear reading (in some cases the results had to be multiplied by 16), of course, magnifies greatly any slight error in the initial observations.

Regarding the use of hydrosulphite as a bleaching agent, many of the chemists reported a difficulty in securing a sufficiently clear reading with the use of this substance alone. There appeared to be an insufficient decolorization in some instances, while in other cases there was a redarkening of the bleached solution due to oxidation. Weisberg^a has recently studied the action of hydrosulphites, and concludes that the action is a double one, first, by means of the free sulphurous acid where the bleaching action is permanent, and secondly, by means of the nascent hydrogen which is evolved, when there is a redarkening of the solution through oxidation. The referee has found that this after-darkening may be prevented by the use of a new hydrosulphite derivative, sodium sulphonylate-formaldehyde, sold commercially as Rongalite C. Hydrosulphite bleaches are being used extensively in sugar work just at present, and the effect of these upon the polarization and reducing power of various sugars are points which should be thoroughly investigated by the referee next year.

The gravimetric determination of the sucrose in the molasses was made by Mr. A. H. Bryan with the following results:

TABLE VII.—Gravimetric determination of sucrose in molasses by Allihn's, and by Munson and Walker's, method, using different clarifying agents.

Clarifying agent.	Allihn method.			Munson and Walker method.		
	Weighing as Cu_2O .	Weighing as CuO .	Titration of Cu by Low's method.	Weighing as Cu_2O .	Weighing as CuO .	Titration of Cu by Low's method.
None.....	<i>Per cent.</i> 33.80	<i>Per cent.</i> 33.98	<i>Per cent.</i> 34.68	<i>Per cent.</i> 33.56	<i>Per cent.</i> 34.19	<i>Per cent.</i> 33.66
Lead subacetate.....	34.63	34.75	35.80	33.56	33.84	34.79
Dry subacetate of lead.....	36.46	34.87	33.68	35.07	33.42	32.41

Centrb1, Zuckerind., 1907, 15: 975.

The determinations of reducing sugars in the sample of molasses are reported in the following table:

TABLE VIII.—*Determination of reducing sugar as dextrose in molasses by Allihn's, also Munson and Walker's, method, using different clarifying agents.*

Clarifying agent and analyst.	Quantity of precipitant used.	Allihn method.			Munson and Walker method.		
		Weighing as Cu_2O .	Weighing as CuO .	Titration of Cu by Low's method.	Weighing as Cu_2O .	Weighing as CuO .	Titration of Cu by Low's method.
None:	cc.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
A. H. Bryan, Washington.....		19.77	19.37	19.45	19.20	18.34	18.43
J. E. Halligan, Louisiana.....		20.68					
W. D. Horne, New York.....		20.60	20.06	19.97	20.00	19.43	19.44
Fritz Zerbán, Louisiana.....		19.39	19.22	19.62	19.12		
Average.....		20.11	19.55	19.68	19.44	18.89	18.94
Subacetate of lead:							
A. H. Bryan, Washington.....	5	17.51	16.47	16.29	17.27	16.26	15.97
J. E. Halligan, Louisiana.....	5	19.55					
W. D. Horne, New York.....	5	19.45	19.16	19.16	19.00	18.53	18.26
Fritz Zerbán, Louisiana.....	5	17.45		17.76	17.87		
Average.....		18.49	17.82	17.74	18.05	17.39	17.12
Dry subacetate of lead:							
A. H. Bryan, Washington.....	a)	17.85	17.55	17.57	17.60	17.32	17.27
J. E. Halligan, Louisiana.....	a)	19.09					
Average.....		18.47	17.55	17.57	17.60	17.32	17.27
Neutral lead:							
J. E. Halligan, Louisiana.....	4	19.99					

a Gram.

VOLUMETRIC METHOD.

Analyst.	Soxhlet method.		Violette method.	
	Clarification.	Dextrose.	Clarification.	Dextrose.
J. E. Halligan.....	None.....	Per cent. 20.40		Per cent.
G. L. Spencer.....			Normal lead..	17.55

The results confirm the observations in the case of sugar, showing that a large amount of reducing sugars is precipitated by basic lead. The slightly higher result obtained in each instance by the Allihn method as compared with that of Munson and Walker is explained by the greater inverting action of the Allihn solution upon the sucrose.

THE EFFECT OF HYDROSULPHITE AND RONGALITE ON THE POLARIZATION OF DEXTROSE, LEVULOSE, AND SUCROSE.

By A. H. BRYAN.

Solutions of 10 grams of the pure sugars in 100 cc of water were prepared and to aliquot parts (25 cc) were added one-fourth, one-half, and 1 gram of the bleaching agents. When these salts were dissolved by shaking, the solutions were made up to 50 cc with water and polarized immediately. The tubes containing the solutions were set aside and polarized after five hours and again

after standing one day. Care was taken to have all the solutions of the same temperature. The results of polarization are contained in the following table:

Determinations of the effect of sodium hydrosulphite B. A. S. F. and rongalite C. on the polarization of dextrose, levulose, and sucrose.

Bleaching agents.	Dextrose.			Levulose.			Sucrose.		
	Imme- diate polari- zation.	After 5 hours.	After 24 hours.	Imme- diate polari- zation.	After 5 hours.	After 24 hours.	Imme- diate polari- zation.	After 5 hours.	After 24 hours.
Original polariza- tion.....	°V. +15.4	°V. +15.3	°V. +15.3	°V. -25.0	°V. -25.0	°V. -25.25	°V. +19.2	°V. +19.2	°V. +19.1
With a crystal or two in polariza- tion tube.....	+15.25	+15.1	+15.0	-25.05			+19.2		
One-fourth gram hydrosulphite.....	+15.1	Cloudy.	+14.85	-25.0	-25.1	-25.35	+19.2	Cloudy.	+18.95
One-half gram hy- drosulphite.....	+15.1	Cloudy.	+14.55	-25.1	Cloudy.	-25.5	+19.1	Cloudy.	+18.9
1 gram hydrosul- phite.....	+15.1	+14.15	+13.75	-25.1	-25.2	-25.5	+19.1	+19.05	+19.05
One-fourth gram rongalite.....	+15.35	+15.3	+15.1	-25.05	-25.2	-25.25	+19.2	+19.05	+19.05
One-half gram ron- galite.....	+15.4	+15.3	+15.1	-25.05	-25.2	-25.3	+19.2	+19.0	+19.05
One gram rongalite.....	+15.4	+15.35	+14.95	-25.1	-25.2	-25.4	+19.1	+19.1	+19.1

Hydrosulphite has more effect on the polarization of the sugars than rongalite, and lowers the polarization of dextrose more than that of the other two sugars. With sucrose there is a change in the immediate polarization on the addition of 0.5 gram, but on standing one day, 0.25 gram reduces the rotation. Whether this action is due to an inversion of the sucrose, it is impossible to say at present. With levulose there seems to be a slight increase in the polarization, but in other experiments with this sugar no change in the rotation was noted. Rongalite has a slight action on the rotation of dextrose, but on sucrose and levulose there is apparently no action.

In regard to bleaching, hydrosulphite is much faster than rongalite, but it has the disadvantage that its decolorizations are not altogether permanent, and on standing a very short time sulphur is precipitated, making the liquid too cloudy for a reading. More work along this line is being done at the Bureau of Chemistry.

THE DETERMINATION OF SULPHUROUS ACID IN MOLASSES.

By FRITZ ZERBAN and W. P. NAQUIN.

At the last meeting of the association, in 1906, W. D. Horne called attention to several sources of errors in the provisional official method for the determination of sulphurous acid in foodstuffs when applied to sugar products. In the meantime, the food law has been enacted, and therefore an investigation into this question could no longer be deferred. The different methods that have heretofore been used were studied and compared. The work has not yet been completed, but it was deemed advisable to present the data collected so far mainly for the information of those who have to make determinations of sulphurous acid in sugar products. The work will be published later in full.

The following questions had to be studied: (1) Whether molasses contains any hydrogen sulphid or gives rise to it when boiled in the presence of free acid; and whether hydrogen sulphid and sulphurous acid can be given off

simultaneously without acting upon each other. (2) Whether there is any error due to outside sources (use of gas for heating, rubber for connections, tin for condensers). (3) Whether any volatile substances are given off during the distillation which are apt to be acted upon by iodine; and whether or not iodine is lost by volatilization. (4) Whether the use of a current of carbon dioxide is necessary. (5) Whether the total amount of sulphurous acid distills over with the first half of the distillate.

According to our present knowledge of the composition of the sugar cane and its products most, if not all, of the organic sulphur is in the form of proteid sulphur. By boiling in an acid solution the proteids are decomposed, and so far the following cleavage products containing sulphur have been isolated: Cystine, cysteine, α - and β -thio-lactic acid, thio-glycolic acid, ethylsulphide, methylmercaptan, and hydrogen sulphide. The thio acids mentioned decompose further upon prolonged heating, yielding hydrogen sulphide. The presence of hydrogen sulphide in the distillation products of molasses is therefore to be expected. This was suggested by W. D. Horne and also by Jerome Alexander in a paper on the determination of sulphurous acid in gelatin. The following experiments were performed to decide this question: Fresh cane juice was boiled down to sirup without the use of any chemicals; 100 grams were diluted to 400 cc, 5 cc of glacial phosphoric acid added, and distilled. The distillate was collected in a flask containing de Koninck's reagent for hydrogen sulphide (mercuric cyanide and ammonium chloride in water). A black precipitate was obtained which after filtration was dissolved in bromine water. The solution gave a precipitate of barium sulphate when tested with barium chloride. Experiments carried out in a similar manner with silver nitrate and lead acetate solutions gave the same results. After receiving a copy of Doctor Horne's paper, cadmium chloride was used and our observations could be confirmed. The filtrate from the cadmium sulphide was also tested for sulphur, with positive results. The volatile sulphur which is not in the form of hydrogen sulphide may be due to the volatilization of substances of a mercaptan-like character. We have not been able so far, on account of the small quantities obtained, to identify them or to prevent their determination together with sulphurous acid.

Similar experiments were performed with a molasses obtained by the sulphitation process as practiced in Louisiana. Hydrogen sulphide was again obtained, which proves that under the conditions of procedure sulphurous acid and hydrogen sulphide may coexist. It is, therefore, advisable to use a solution of cadmium-chloride in the determinations, as recommended by Horne.

Gas can be used for heating the distilling flask without any risk. But if it be found necessary to concentrate the distillate, this should be done in flasks with narrow openings. Rubber connections should be avoided as much as possible, especially at that side of the condenser which is connected with the receiver containing iodine or bromine. It was found that the use of rubber may introduce considerable error. When two glass tubes are to be connected with rubber hose the two ends of the glass tubes should touch. In a number of distillations a Kjeldahl condenser with tin serpentines was used, and in this case a slight deposit of sulphur was invariably noticed in the glass tube extending into the receiver. When glass condensers were substituted for those of tin that deposit could not be observed. The deposit is probably due to the interaction of hydrogen sulphide and sulphur dioxide, but the tin seems to play some part in this reaction.

Doctor Horne called attention to the possibility that sugar products like molasses when distilled in acid solution might give off volatile organic substances which are acted upon by iodine. In order to test this question, a sample

of molasses was secured from a house where no sulphur is used. In one experiment the distillate was collected in bromin water after washing with cadmium-chlorid solution, and the sulphur determined gravimetrically in the cadmium sulphid as well as in the filtrate from it combined with the bromin water. In a second determination the official method was used, the dilution in the distilling flask and the quantity of liquid distilled over being the same as in the first experiment. It was found that the official method yielded considerably more "sulphur" than was obtained by the gravimetric determination. However, this discrepancy is partly due to the fact that in spite of the U tube provided for in the official method some iodine volatilizes during the distillation. This could easily be shown by connecting the U tube with a glass tube extending into a small flask which contained a solution of potassium iodide. When the distillation was finished the potassium iodide had taken up a considerable quantity of iodine, not enough, however, to make up for the difference from the gravimetric method.

Several experiments conducted with and without the use of a current of carbon dioxide proved the necessity of using this precautionary measure, although the influence was not so great as we had anticipated. The inlet tube for the carbonic acid gas must extend into the liquid to prevent it from distilling back into this same tube.

After these preliminary experiments the following mode of procedure was adopted, avoiding the errors recognized so far: Transfer 50 grams of molasses to a flask of sufficient capacity and make up to 400 cc with air-free water. Close the flask with a rubber stopper (rubber can safely be used at this point) having three perforations. One of these is for the inlet tube for the carbon dioxide, the second for a small separatory funnel filled with glacial phosphoric acid, the third for a catch-all connected by means of a good cork with a Liebig condenser. Connect the other end of the condenser with a glass tube leading into a filtering flask containing a small quantity of a 2 per cent solution of cadmium chloride. The glass tube must terminate below the surface of the liquid. After the filtering flask follows a U tube containing a little water and then a glass tube leading into bromin water in a little Erlenmeyer flask. The U tube prevents sucking back of the bromin water into the cadmium solution. After the receivers have been put in place, carbon dioxide is passed through the entire apparatus for about ten minutes. Then about 5 cc of phosphoric acid are added from the separatory funnel and 200 cc of the liquid distilled over. When the distillation is finished, the bromin water in the Erlenmeyer and the contents of the U tube are transferred to a beaker, and the cadmium solution containing the distillate is filtered into the bromin water. The excess of bromine is boiled off and the sulphuric acid is precipitated as barium sulphate and weighed. If the hydrogen sulphide is to be determined also, the cadmium sulphide is dissolved in bromin water and the solution treated like the others.

It was deemed inadvisable to distil a smaller volume than 200 cc in order to avoid charring of the mass at certain points and subsequent reduction of sulphuric acid.

Finally the influence of concentration was studied and very surprising results obtained. When the same quantity of molasses was diluted in one case to 600 cc and in the other to 400 cc, both being distilled down to 200 cc, the first experiment gave a much higher result than the second. This experience led us to investigate how much of the liquid must be distilled to obtain the entire quantity of SO_2 . It was found that by adding again 200 cc of water after having distilled from 400 to 200 cc new quantities of sulphurous acid were obtained, and it was necessary to repeat this procedure four times to distil

over practically all of the SO_2 . In this case the last distillate contained only traces of sulphur. It was thus shown that by distilling over 800 cc the SO_2 could be satisfactorily determined. The method would be facilitated by making up 50 grams of molasses to 1,000 cc and distilling down to 200 cc. In this case the receivers had naturally to be changed to some extent. Results agreed practically with those obtained by separate distillations. After the true amount of SO_2 in a certain molasses had been determined in this way, the official method was tested for its accuracy. By distilling in the same way from 1,000 cc to 200 cc of a N/10 iodine solution for the collection of the distillate and titrating with thiosulphate, results considerably higher than those by the above method were obtained.

The necessity of distilling off 800 cc of liquid renders the method very tedious, and it would be desirable to devise one which requires less time and labor. It is possible that the sulphurous acid may be determined in some other way than distillation. Direct titration is impossible on account of the dark color of the products under examination, but some method of precipitation may directly or indirectly accomplish the purpose. It would also be desirable to find some method by which the different forms of sulphur dioxide in molasses could be distinguished.

Mr. Wiley called attention to the value and timeliness of Mr. Zerban's paper, inasmuch as both State and Federal food officials are concerned with the difficulties in distinguishing between natural sulphur and added sulphites.

Upon motion by Mr. Wiley, election of officers was made special order for 10 o'clock Friday; and on motion by Mr. Van Slyke reports of all special committees, except the committees on recommendations of referees, were made special order for 11 o'clock.

The association adjourned for the day.

THIRD DAY.

FRIDAY—MORNING SESSION.

REPORT ON MEDICINAL PLANTS AND DRUGS.

By L. F. KEBLER, *Referee.*

The burden of other work imposed upon the staff of the drug laboratory and other pharmaceutical chemists of the country in consequence of recent food and drug legislation, both National and State, has interfered somewhat with the prosecution of the cooperative work on drug assaying during the past year. It is hoped that when the most urgent problems have been solved the study of methods will receive an amount of attention commensurate with its fundamental importance. In the meantime it has been deemed advisable not to depend upon the cooperation of chemists generally in the work, but to continue the same within the drug laboratory as circumstances permit until the time is more propitious for general participation.

In view of the legal status conferred upon the United States Pharmacopœia, Eighth Revision, it seemed desirable to study all of the plant drug assays official therein, comparing them with some of the best available nonofficial methods. After this work was begun, the committee of revision of the Pharmacopœia promulgated two lists of additions and corrections under date of May 1 and June 1, 1907, which introduced some minor changes in the details of certain methods. These changes were recognized in the work subsequently done.

The work this year covered methods for the assay of aconite leaves, aconite root, belladonna leaves, belladonna root, cinchona bark (yellow and red), coca leaves, colchicum corm and colchicum seeds. Samples of these drugs, delivered as being of United States Pharmacopœia quality, were made the basis of study, and the following programme of work was pursued by three collaborators in the drug laboratory. All calculations and solutions, except as otherwise specified, were based on the data of the United States Pharmacopœia, Eighth Revision.

DIRECTIONS FOR THE WORK.

VOLUMETRIC SOLUTIONS.

Prepare a standard normal sulphuric acid, determining the factor by two or more of the following methods:

(1) The United States Pharmacopœia method by titration with normal potassium hydroxid solution which has been standardized against purified potassium bitartrate.

(2) By titration of the ignition residue from a weighed portion of potassium bitartrate.

(3) By titration of a weighed amount of freshly ignited sodium carbonate.

(4) By precipitation as barium sulphate and gravimetric determination.

(5) By evaporation with slight excess of ammonium hydroxid, and determination after drying at 125° C. as ammonium sulphate.

From this normal acid prepare a decinormal sulphuric acid by dilution.

Prepare a fiftieth normal solution of potassium hydroxid and determine the factor by titration with the decinormal sulphuric acid prepared as just directed.

DETERMINATION OF ALKALOID.

Total extraction method.

Into a 200 cc flask weigh 10 grams of the powdered drug, add about 75 cc of ether-chloroform mixture (5 to 1 by volume), rotate and add 5 cc of a mixture

of 40 cc of strong ammonia water with 60 cc of alcohol, cork, shake well and often during one hour, and let stand over night. Transfer as much of the mixture as possible to a small percolator, the neck of which is provided with a purified cotton plug, receiving the percolate in a beaker or flask. Rinse the residue in the flask into the percolator with additional portions of the ether-chloroform mixture, packing it down moderately with a glass rod, and continue the percolation with the same mixture until the percolate does not give an alkaloidal reaction, or at most only an opalescence when 2 cc are evaporated, and the residue is treated with N/10 hydrochloric acid, the mixture filtered and the filtrate tested with Mayer's solution. Evaporate or distil the percolate to about 10 cc at a temperature not exceeding 70° C. Transfer to a separator, and rinse the vessel with two portions of 20 cc each of ether, alternating with two portions of 10 cc each of 2 per cent sulphuric acid. Shake out, making sure that the reaction is acid, and repeat the operation three times with 15 cc of the same strength of acid, filtering the acid solutions into another separator through a 7 cm filter, and rinsing the latter with a few cubic centimeters of water. Wash the acid solution with 10 cc of ether-chloroform mixture (1 to 3 by volume), discarding the latter, and if colored repeat until no more color is acquired. Make the solution alkaline with ammonia water and shake out with four successive portions of about 20 cc each of ether-chloroform mixture (1 to 3); collect the latter in a separator, wash with 5 cc of water, transfer the ether-chloroform mixture to a tared flask or beaker, rinsing the separator with a small portion of ether-chloroform, and evaporate the solvent at a temperature not exceeding 70° C.

(a) *Gravimetric.* Add 3 cc of ether, evaporate and dry to constant weight at a temperature not exceeding 70° C. Report weight of alkaloidal residue.

(b) *Volumetric.* Dissolve the residue in about 10 cc of neutral alcohol, add 25 cc of water, 3 to 15 drops of cochineal indicator solution, measure in from a burette a slight excess of N/50 sulphuric acid, mix intimately, and titrate back with N/50 potassium hydroxid. Report net amount of N/50 acid required.

Aliquot method.

Into a 200 cc flask weigh 15 grams of the powdered drug, add 150 cc of ether-chloroform mixture (5 to 1 by volume), cork and shake often for several minutes. Add 10 cc of ammonia water (10 per cent), shake frequently during one hour, and let stand over night. Add 15 cc of water, or sufficient to agglomerate the drug, shake, let settle a few minutes, and then decant 75 cc of the clear solution into a graduated cylinder. Transfer the solution to a separator, rinsing the cylinder with a few cubic centimeters of ether-chloroform, and shake out with 20 cc (or sufficient to render acid) of 2 per cent sulphuric acid, repeating the operation three times with 15 cc of the same strength acid, and collect the acid solutions in another separator. Wash the acid solution with 10 cc of ether-chloroform mixture (1 to 3 by volume), discarding the latter, and, if colored, repeat until no more color is acquired. Make the solution alkaline with ammonia water, and shake out with four successive portions of about 20 cc each of ether-chloroform mixture (1 to 3); collect the latter in a separator, wash with 5 cc of water, transfer the ether-chloroform mixture to a tared flask or beaker, rinsing the separator with a small portion of ether-chloroform, and evaporate the solvent at a temperature not exceeding 70° C.

(a) *Gravimetric.* Add 3 cc of ether, evaporate and dry to constant weight at a temperature not exceeding 70° C. Report weight of alkaloidal residue.

(b) *Volumetric.* Dissolve the residue in about 10 cc of neutral alcohol, add 25 cc of water, 5 to 15 drops of cochineal indicator solution, measure in from a burette a slight excess of N/50 sulphuric acid, mix intimately, and titrate back with N/50 potassium hydroxid. Report net amount of N/50 acid required.

Whenever iodeosin indicator is used, follow the directions of the United States Pharmacopœia VIII, page 542. Solutions standardized by means of cochineal should not be used with iodeosin without running a blank for comparison, as the indicators do not give the same neutral point.

These methods and those referred to in the United States Pharmacopœia are applied to the analysis of the following drugs with the modifications noted.

ACONITE ROOT.

Method I.

U. S. Pharmacopœia VIII, p. 28. Report the amount of N/10 acid required.

Method II.

Alliquot gravimetric. Use *ether* instead of ether-chloroform mixture in the final shaking out.

BELLADONNA LEAVES.

Method I.

U. S. Pharmacopœia VIII, p. 67. Report indicator used and amount of N/10 acid required.

Method II.

Alliquot volumetric.

BELLADONNA ROOT.

Method I.

U. S. Pharmacopœia VIII, p. 68. Report indicator used and amount of N/10 acid required.

Method II.

Alliquot volumetric.

CINCHONA BARK.

Method I.

U. S. Pharmacopœia VIII, p. 102. Report total and ether-soluble alkaloids.

Method II.

Total extraction, gravimetric.

COCA LEAVES.

Method I.

U. S. Pharmacopœia VIII, p. 106. Report indicator used and amount of N/10 acid required.

Method II.

Alliquot gravimetric. Use *ether* instead of ether-chloroform mixture throughout.

COLCHICUM CORM.

Method I.

U. S. Pharmacopœia VIII, p. 111. Report weight of alkaloidal residue.

Method II.

Exhaust 10 grams of the powdered drug by percolation with alcohol, add 25 cc of water to the solution and evaporate. Digest and stir the residue for several minutes with 25 cc of petroleum ether and transfer the liquid to a beaker, rinsing the residue with a little fresh petroleum ether. Add to the beaker 20 cc of water and evaporate the petroleum ether. Gently warm the first residue with 10 cc of water, cool, and filter the solution through a small filter into a separator, rinsing the vessel, and filter with several small portions of water and finally with the water in the beaker. Shake out the liquid with four portions of 10 cc each of chloroform and evaporate the latter in a tared dish. Add 3 cc of alcohol and evaporate, repeat the operation and dry the residue to constant weight at a temperature not exceeding 70° C. Report weight of alkaloidal residue.

COLCHICUM SEED.

Method I.

U. S. Pharmacopœia VIII, p. 112. Report weight of alkaloidal residue.

Method II.

See Colchicum Corm. Method II.

STANDARDIZATION OF VOLUMETRIC SOLUTIONS.

In order to compare the present pharmacopœial method for preparing volumetric solutions, a quantity of potassium bitartrate was purified according to the prescribed directions of the Pharmacopœia. Two equal parts of this purified potassium bitartrate were weighed out, one portion ignited at a low red temper-

ature in a platinum dish, dissolved in water, and carefully mixed with the second portion weighed as above. The resulting mixture was acid in reaction to phenolphthalein, indicating that some of the bitartrate was possibly lost during the ignition. Several repetitions of the above indicated that there apparently was some loss on ignition and that the method indicated in the Pharmacopœia required further study. Recourse was then had to normal sulphuric acid prepared by the usual methods, titrated by two independent workers using several indicators, with satisfactory results, the variations ranging from the lowest for methyl orange, 0.9890, to the highest for phenolphthalein, 1.0065. This solution was used as the basis for preparing decinormal sulphuric acid.

Mr. Parker's portion of the work was practically completed when the pharmacopœial corrections appeared, and his results are therefore based upon the original methods, except that cochineal had been substituted for hematoxylin. The standard acid employed by him, however, was found to correspond closely with that employed by the other analysts.

The results obtained are given in the following table:

Comparison of methods for the assaying of aconite, belladonna, cinchona, coca leaves, and colchicum.

Analyst.	Aconite leaves.			Aconite root.		
	Method I, U. S. P.	Method II, aliquot.		Method I, U. S. P.	Method II, aliquot.	
		Gravi- metric.	Volu- metric.		Gravi- metric.	Volu- metric.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Parker.....	0.243	0.241	0.179	0.486	0.588	0.410
Do.....	.256	.245	.154	.499	.447	.384
Do.....						
Rieger.....	.173	a. 234	a. 173	.499	.517	
Do.....	.173			.416	.528	
Do.....					.516	
Do.....						
Warren.....	.167	.231		.335	.612	.610
Do.....	.172	.254		.346	.611	
Do.....	.157					
Average.....	.191	.241	.169	.430	.546	.468
Maximum.....	.256	.254	.179	.499	.612	.610
Minimum.....	.157	.231	.154	.335	.447	.384
Difference.....	.099	.023	.025	.164	.165	.226

Analyst.	Belladonna leaves.			Belladonna root.		
	Method I, U. S. P.	Method II, aliquot.		Method I, U. S. P.	Method II, aliquot.	
		Gravi- metric.	Volu- metric.		Gravi- metric.	Volu- metric.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Parker.....	0.344	0.362	0.229	0.494	0.530	0.384
Do.....	.356	.409	.224	.488	.516	.384
Do.....	.356					
Rieger.....	.287		.253	.406		.423
Do.....	.244		.240			
Do.....	.270			.407		
Do.....	.270					
Warren.....	.242		.264	.466		.403
Do.....	.284		.254	.460		.387
Do.....						
Average.....	.295	.386	.244	.442	.523	.396
Maximum.....	.356	.409	.264	.494	.530	.423
Minimum.....	.242	.362	.224	.370	.516	.384
Difference.....	.114	.047	.040	.124	.014	.039

* Mean of two assays.

Comparison of methods for the assaying of aconite, belladonna, etc.—Continued.

Analyst.	Cinchona (yellow).					Cinchona (red).				
	Method I, U. S. P., alkalo- loid.		Method II, total ex- traction alkaloid.			Method I, U. S. P., alkalo- loid.		Method II, total ex- traction alkaloid.		
	Total.	Ether soluble.	Total.	Ether soluble.	Marc.	Total.	Ether soluble.	Total.	Ether soluble.	Marc.
	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.
Parker.....	8.11	6.72	8.52		0.26	5.90	4.10	5.06		
Do.....	8.02	6.55	8.41		.08	5.27	4.27	4.91		
Do.....	8.24	6.81	8.08		.59					
Rieger.....	7.85	6.07	7.11	4.45	.30	5.64	3.64	5.40	3.05	0.31
Do.....	8.20	4.93	6.85	4.81	.49	5.73	3.25	5.32	3.01	.18
Warren.....	8.52	5.83	8.26	5.37	.40	6.48	3.51	5.38	3.41	.59
Do.....	8.75	4.98	7.53	5.09	.43	5.56	2.86	5.68	3.46	.35
Do.....	8.79	5.34	7.03	4.67	.28					
Average.....	8.31	5.90	7.72	4.88	.35	5.59	3.61	5.29	3.23	.36
Maximum.....	8.79	6.81	8.52	5.37	.59	5.90	4.27	5.68	3.46	.59
Minimum.....	7.85	4.93	6.85	4.45	.08	5.27	2.86	4.91	3.01	.18
Difference.....	.94	1.88	1.67	.92	.51	.63	1.41	.77	.45	.41

Analyst.	Coca leaves.			Colchicum corm.		Colchicum seeds.	
	Method I, U. S. P.	Method II, aliquot.		Method I, U. S. P.	Method II.	Method I, U. S. P.	Method II.
		Gravi- metric.	Volu- metric.				
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Parker.....				0.454	0.275	0.940	0.611
Do.....	0.498	0.738	0.558	.39	.283	.940	.597
Do.....	.510	.708	.540	a (.39)		a (.392)	
Rieger.....				.354	.282	.846	.496
Do.....	.613	.724	.592	.334	.260	b (.532)	b (.411)
Do.....	.569	.700	.626			1.044	1.190
Do.....						b (.526)	b (.452)
Warren.....	.382	.643	.400	.316	.286	.642	.658
Do.....	.407	.592	.348	.290	.298	.722	.588
Average.....	.496	.684	.511	.356	.281	.856	.690
Maximum.....	.613	.738	.626	.454	.298	1.044	1.190
Minimum.....	.382	.592	.348	.290	.260	.642	.496
Difference.....	.231	.146	.278	.164	.038	.402	.694

* Residue purified by re-solution in ether, treatment with water, etc., as in the assay of colchicum corm, U. S. P., not included in average, maximum, or minimum.

* Residue purified as above, substituting petroleum ether for ether, not included in average, maximum, or minimum.

DISCUSSION OF RESULTS.

ACONITE LEAVES.

The leaves were assayed by the same methods as the root. The variation by the United States Pharmacopœia method reached 51 per cent of the average value. The aliquot gravimetric method gave more uniform results, the variation amounting to 10 per cent; but titration in a few cases indicated that, as is frequently the case, the gravimetric results are high. The Squibb physiological test was obtained with a dilution of 1-80. It will be noted that the indications by this test and by assay do not correspond to the analogous results for aconite root.

ACONITE ROOT.

For aconite root, as for the leaves, the aliquot gravimetric results showed less variation than the United States Pharmacopœia method, but the few titration results indicate that some of the former are considerably too high. The

Squibb physiological test gave a value of 1-500, or six times as great as the leaf, instead of two to three times, as might be inferred from the assays.

BELLADONNA LEAVES.

The results of Mr. Parker by the United States Pharmacopœia method for belladonna leaves are decidedly higher than those of the other analysts, which do not vary much among themselves. The discrepancy may indicate some deterioration in the drug in the three months intervening before the lower results were obtained. All the aliquot volumetric results, which were obtained about the same time, are fairly concordant. Experiments were made to determine whether, in the extraction of the drug by ethereal solvents, the presence of a small amount of water exercises an unfavorable influence. In the pharmacopœial method a few cubic centimeters are introduced in the form of diluted ammonia water. Mr. Parker varied the method by using alcoholic ammonia of corresponding strength (about 4 per cent), also by diluting stronger ammonia water with alcohol instead of water to the same strength. The results were decidedly lower, namely, 0.298 and 0.276 per cent.

Mr. Warren employed the total extraction method also, which is similar to the pharmacopœial method, except in the use of alcohol for diluting the ammonia. He obtained higher gravimetric results, but titration of the residues indicated that the increase was due to impurities.

Mr. Rieger tried the total extraction method, using alcohol in one test and water in another, to dilute the ammonia water. His results are low in both cases, and no conclusions can be drawn except that the residues are impure.

BELLADONNA ROOT.

In the results for belladonna root, as in those for the leaves, the best agreement is obtained by the aliquot volumetric method, and the gravimetric results are evidently vitiated by impurities. Just as with belladonna leaves, the results of experiments on the influence of small amounts of water on the extraction of the root led to no conclusion. No positive advantage was shown to accrue from the substitution of alcohol.

CINCHONA (YELLOW AND RED).

The pharmacopœial method for cinchona gave much less variation in the results on total alkaloid than the total extraction method, but, as was the experience last year, the results on ether-soluble alkaloid are not concordant.

The change in the pharmacopœial method, doubling the amount of solvent used in extracting the drug, involves the shaking out of 200 cc of ether-chloroform mixture with comparatively small volumes (15, 10, and 5 cc) of dilute acid, which is a tedious and difficult task. Evidently some nonalkaloidal matter passes into the acid solution in shaking out, and from that into the alkaloidal residue, which is discolored in all cases.

In determining ether-soluble alkaloid Mr. Warren filtered the ethereal solution through cotton in a small funnel.

The total extraction method fails to exhaust the marc even after obtaining 400 to 500 cc of percolate, though weak runnings give no test for alkaloids. On testing the marc, as directed last year, by boiling with normal hydrochloric acid and shaking out the acid extract, the amounts of impure alkaloid indicated in the column under "marc" were obtained.

COCA LEAVES.

The results by all methods for coca leaves are so variable that none can be said to be encouraging. The results of the aliquot gravimetric method agree best among themselves, but the titration equivalents show that they must be regarded as too high.

COLCHICUM CORM.

The results for colchicum corm by the pharmacopœial method are materially higher than by Method II, but also vary relatively more among themselves.

COLCHICUM SEEDS.

Results for colchicum seeds, by both methods, exhibit great variations, and experiments on purifying the residues by a process similar to the latter part of the pharmacopœial method for colchicum corm showed that the impurities present would render the results entirely unreliable unless they were removed.

In the absence of the referee on tannin, his report was presented by Mr. Tolman, as follows:

REPORT ON TANNIN.

By F. P. VEITCH, *Referee*.

The referee was unable to obtain the samples desired until very late, and for this reason very few of the twelve or fifteen analysts, who had promised to collaborate, were able to do so and the work was confined to a comprehensive study of the filtration of soluble solids through folded S and S 590 and S and S 588 paper, the first a high-grade paper and the latter a cheaper paper of ordinary grade. For a number of years the No. 590 paper has been unsatisfactory for use in soluble-solids determinations, as it is not uniform and is too weak to stand the weight of the solution after the paper is folded.

In a study which the referee had made, using a large number of different extracts, it was found that the No. 588 paper gave results agreeing closely with those with the No. 590 paper, except in the case of quebracho. Reed^a had previously obtained satisfactory results with No. 588, but only on hemlock and lentiscus. The referee's favorable results led him to submit the paper to collaborative tests, as both time and expense would be saved if it could be used. The following circular letter was sent out:

DIRECTIONS FOR TANNIN WORK, 1907.

DEAR SIR: I am forwarding you to-day three samples of extracts in which the following determinations are to be made in triplicate:

- (1) Total solids.
- (2) Soluble solids, using 15 cm 590 S and S folded filters.
- (3) Soluble solids, using 15 cm 588 S and S folded filters.

Make all dryings on the bottom shelf of a boiling-water oven, drying for 12 hours.

Dissolve the following quantities of extracts in 2,700-cc of water at 80° C: Sample No. 1, 18 grams; Sample No. 2, 42 grams; Sample No. 3, 15 grams.

Allow to cool overnight to 20° and dilute to 3,000 cc. In these solutions determine total and soluble solids as follows:

Total solids: Thoroughly mix the solution, immediately pipette 100 cc into a tared dish, evaporate, and dry as directed.

^a J. Soc. Chem. Ind., 1902, 21: 691.

Soluble solids: Add 75 cc of solution (kept at from 20° to 25° C during filtration) to 2 grams of kaolin (free from soluble salts); stir, let stand 15 minutes, decant, and discard as much as possible of the supernatant liquid. Again add 75 cc of the tannin solution to the kaolin, stir, and pour immediately on a folded filter. Keep the filter full and the funnel and receiving vessel covered. Reject the first 150 cc of filtrate, evaporate and dry the next 100 cc (which must be as clear as practicable) as directed. * * *

If you have not the kind and size of filter paper specified please communicate with the referee before proceeding further.

The results reported are given in the following table:

TABLE I.—*Determinations of total and soluble solids in three extracts, comparing S and S 588 and 590 filter papers.*

Analyst.	Temperature.	Total solids.			Soluble solids.			
		Sample 1.	Sample 2.	Sample 3.	Sample 1.			
					S and S 590 filter paper.	Time. ^a	S and S 588 filter paper.	Time. ^a
	° C.	Per cent.	Per cent.	Per cent.	Per cent.	Minutes.	Per cent.	Minutes.
F. C. Sammett.....	25	85.8	45.9	89.5	80.0	32-42	79.6	35-45
C. C. Smoot.....	26	84.4	45.2	88.3	78.7	54-81	78.5	56-85
Do.....	20	84.8	45.5	88.7	77.8	52-82	77.6	55-80
F. P. Veltech.....	24	85.1	45.5	88.8	80.2	95-140	79.3	90-110
Chas. Eachus.....		84.4	45.2	88.2	78.3		76.3	
A. G. Stillwell.....		87.7	45.3	90.6	82.5		79.8	

Analyst.	Temperature.	Soluble solids.							
		Sample 2.				Sample 3.			
		S and S 590 filter paper.	Time. ^a	S and S 588 filter paper.	Time. ^a	S and S 590 filter paper.	Time. ^a	S and S 588 filter paper.	Time. ^a
	° C.	Per ct.	Min.	Per ct.	Min.	Per ct.	Min.	Per ct.	Min.
F. C. Sammett.....	25	44.6	34-38	44.5	37-40	88.3	35-30	87.8	33-25
C. C. Smoot.....	26	44.4	33-39	44.2	30-35	87.8	21-23	87.4	25-23
Do.....	20	42.0	70-150	42.5	54-115	87.9	27-23	87.7	30-24
F. P. Veltech.....	24	44.4	48-52	44.3	46-45	87.9	30-31	87.5	32-22
Chas. Eachus.....		44.2		43.6		87.2		86.8	
A. G. Stillwell.....		43.6		43.3		89.2		88.6	

^a Under "Time" the first figures show the time required for the first 150 cc; the second figures for the next 100 cc.

No final conclusion can be drawn from these few results, but it is quite evident that the S and S 588 paper gives slightly lower results than the 590 paper under the conditions of filtration, which, it must be remembered, were devised for use with the 590 paper. The results agree in general with those that have been previously obtained, and it seems probable that with some modification in the method of filtration, such as the filtration of about 200 cc before collecting the portion for analysis, the results would agree with those obtained with the 590 paper. Such a procedure would, of course, lengthen the time of filtration, and this would not be desirable.

The time of filtration with the 588 paper was, as a rule, slightly shorter than with the 590 paper. Mr. Smoot observed that the temperature at which filtration was conducted affected the speed of filtration as well as the percentage of soluble solids; the cooler the solution the more slowly it filtered. Mr. Smoot also made some determinations with an asbestos-kaolin mat on a Buckner fun-

nel, using suction as proposed by Reed at the recent meeting of the Association of American Leather Chemists. The results were as follows:

TABLE II.—*Determination of soluble solids in tannin extracts using an asbestos-kaolin mat, at 21° C.*

Sample 1.		Sample 2.		Sample 3.	
<i>Per cent.</i>	<i>Minutes.</i>	<i>Per cent.</i>	<i>Minutes.</i>	<i>Per cent.</i>	<i>Minutes.</i>
77.2	11-26	41.7	11-48	87.8	4-8
77.4	12-28				
78.1	15-45	41.6	20-52	88.4	4-3
77.4	20-68				
78.6	20-68	41.7	21-42		

The first figure under "Minutes" shows the time required for the first 150 cc; the second figure for the next 100 cc, with the exception of the first two results on Sample 1 and both results for Sample 3 which were obtained after filtering only 100 cc in each case.

As will be seen, the time required for filtration when the asbestos mat was used was greatly reduced. When the quantity of liquor rejected was at least 150 cc, the results agreed fairly well with those obtained with S and S 590 paper, except on Sample 2 which gave exceptionally clear filtrates with the asbestos filter. This may account for the low results.

The referee does not recommend a change of papers for filtering soluble solids, as the results obtained with 588 paper, while usually sufficiently close to those by the 590 paper, are apt to be wide on quebracho. Doubtless the 588 paper could be safely used on tannery liquors and other materials of low tannin content.

RECOMMENDATIONS.

It is recommended—

(1) That the work on the filtration of tannin extracts be continued with a view to finding a cheap, strong paper as a substitute for S and S 590.

(2) That the method of filtering soluble solids suggested by Reed be given further trial.

(3) That the referee take up the study of methods for the analysis of leather, as such methods are greatly needed.

REPORT ON SOILS.

By J. H. PETTIT, *Referee.*

The work of the referee on soils for the present year has been mainly a continuation of that undertaken last year. Two of the four samples used were the same as two of those sent out last year, while Samples 1 and 3 were new. The following outline of the work to be done was sent to those desiring to participate:

(a) Determine dry matter upon 5 grams of the air-dried soil by heating five hours in water oven.

(b) Make a preliminary digestion of the air-dried soil in fifth-normal nitric acid at room temperature.^a Weigh 200 grams of soil into a 2½ liter

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 46, Revised, p. 74 (b); Bul. 107, p. 18 (b).

flask and add 2,000 cc of a solution of nitric acid of such strength that, after allowing for the nitric acid neutralized by the soil, as shown by the previous digestion, there will be left 2,000 cc of fifth-normal nitric acid solution. Digest five hours at room temperature, shaking every half hour. Record the temperature of digestion. At the end of the period shake and filter through a large folded filter, pouring the solution back into the filter until it runs through clear.

Evaporate 1,000 cc of the filtrate to dryness, moisten with hydrochloric acid, dry again, heat one hour at 110° C to insure complete removal of acid, take up with hydrochloric acid and water, heating if necessary, filter out silica, and wash. Evaporate filtrate and washings to about 10 cc. Add 5 cc of concentrated nitric acid, neutralize with ammonia, make barely acid with nitric acid, heat to from 40° to 50° C, precipitate with ammonium molybdate solution,^a and let stand over night. Filter, wash with 0.1 per cent ammonium nitrate solution until free of acid, and twice with cold water. Dissolve in a standard potassium hydroxid solution, 1 cc of which contains 8.324 mg of potassium hydroxid and is equivalent to 0.2 mg of phosphorus. Titrate the excess with a nitric acid solution, 1 cc of which is equivalent to 1 cc of the potassium hydroxid solution.

(c) Weigh 10 grams of sodium peroxid into an iron or porcelain crucible and thoroughly mix with it 5 grams of soil; if the soil is very low in organic matter add a little starch to hasten the action. Heat the mixture carefully by applying the flame of a Bunsen burner directly upon the surface of the charge and the sides of the crucible until the action starts. Cover crucible until reaction is over, and keep at a low red heat for twenty-five minutes; do not allow fusion to take place. By means of a large funnel and a stream of hot water transfer the charge to a 500 cc measuring flask. Acidify with hydrochloric acid and boil; let cool and make up to the mark. If the action has taken place properly there will be no particles of undecomposed soil in the bottom of the flask. Allow the silica to settle and draw off 200 cc of the clear solution.

Precipitate the iron, aluminum, and phosphorus from the hot solution with ammonium hydroxid; do not allow to settle; filter; wash three or four times with hot water; return the precipitate to the beaker with a stream of water, holding the funnel over the beaker, and dissolve the precipitate in hot dilute hydrochloric acid, pouring the acid upon the filter to dissolve any precipitate remaining. Remove silica and determine phosphorus as in (b). Determine phosphorus, using the alkali carbonate fusion method^b and getting rid of the silica as suggested above.

(d) Fuse 1 gram of soil according to the well-known J. Lawrence Smith method. Transfer the fused mass to a porcelain dish, slake with hot water, grind finely with an agate pestle, and transfer to a filter. After washing free of chlorids, concentrate the filtrate and washings in a Jena beaker to about 20 cc and filter. Slightly acidify filtrate and washings with hydrochloric acid, concentrate in a platinum dish, and add 1½ cc of a platinum chlorid solution (10 cc contains 1 gram of platinum). Evaporate to a sirupy consistency as usual and wash with 80 per cent alcohol and an ammonium chlorid solution. Determine potassium according to the regular J. Lawrence Smith method.^c

The following laboratories sent reports:

S. D. Averitt, for the Kentucky Experiment Station.

H. D. Edmond, for the Connecticut Experiment Station.

R. W. Thatcher, for the Washington Experiment Station.

G. S. Fraps, for the Texas Experiment Station.

J. H. Norton, for the Arkansas Experiment Station.

J. H. Pettit, for the Illinois Experiment Station.

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 46, Revised, p. 11 (b); Bul. 107, p. 2 (c).

^b Fresenius's Quantitative Chemical Analysis, p. 422.

^c Ibid, p. 426.

TABLE I.—*Determination of phosphorus soluble in fifth-normal nitric acid.*

[Parts per million in dry soil.]

Analyst.	Temperature of extraction.	Soil No. 1.	Soil No. 2.	Soil No. 3.	Soil No. 4.
	° C.				
S. D. Averitt, Kentucky.....	29	{ 15.4 11.4		187.4	{ 185.0 187.0
H. D. Edmond, Connecticut ^a	25	10.3	8.2	182.6	188.2
H. R. Watkins, Washington.....		8.8	7.3	185.0	167.2
J. H. Norton, Arkansas.....		{ 14.9 15.1	{ 12.7 10.0	{ 187.2 189.4	{ 177.0 169.0
A. Ystgard, Illinois.....	25	12.7	10.0	188.3	175.2
S. E. Asbury, Texas ^a	30	26.0		169.0	166.2
E. Carlyle, Texas ^a	30	27.5		393.0	

^a Duplicates not reported.TABLE II.—*Determination of phosphorus by the sodium peroxid fusion.*

[Percentage of dry soil.]

Analyst.	Soil No. 1.	Soil No. 2.	Soil No. 3.	Soil No. 4.
S. D. Averitt, Kentucky.....	{ 0.043 .041		0.095	0.103
H. D. Edmond, Connecticut ^a	.045	0.055	.101	.099
J. H. Norton, Arkansas.....	{ .046 .044	{ .065 .059	.108	.101
A. Ystgard, Illinois.....	{ .047 .046	{ .051 .052	.108	.108
A. W. Gregory, Illinois.....	{ .040 .040	{ .100 .102	.094	.094
J. H. Pettit, Illinois.....	.096	.096	.094	.082
			{ .096 .094	{ .090 .088

^a Duplicates not reported.TABLE III.—*Determination of phosphorus by alkali carbonate fusion.*

[Percentage of dry soil.]

Analyst.	Soil No. 1.	Soil No. 2.	Soil No. 3.	Soil No. 4.
S. D. Averitt, Kentucky ^a	0.035		0.092	0.097
A. Ystgard, Illinois.....	{ .046 .046	{ 0.052 .052	.103	.100
			.105	.100

^a Duplicates not reported.

It is unfortunate that no more work by the standard method was done. The results obtained by the sodium peroxid method show fair agreement with the meager data obtained by the standard alkali carbonate method.

TABLE IV.—*Determination of total potassium by the regular method.*

[Percentage of dry soil.]

Analyst.	Soil No. 1.	Soil No. 2.	Soil No. 3.	Soil No. 4.
H. D. Edmond, Connecticut ^a	0.461	0.590	0.477	0.572
H. R. Watkins, Washington.....	{ 1.227 1.381	{ 1.790 1.620	{ 2.449 2.395	{ 1.732 1.719
A. Ystgard, Illinois.....	{ 1.453 1.438	{ 1.852 1.838	{ 1.771 1.765	{ 1.826 1.808

^a Reported unsatisfactory, but no time to repeat.

TABLE V.—*Determination of total potassium by modified method.*

[Percentage of dry soil.]

Analyst.	Soil No. 1.	Soil No. 2.	Soil No. 3.	Soil No. 4.
S. D. Averitt, Kentucky ^a	1.250		1.270	1.465
A. Ystgard, Illinois.....	1.449	1.846	1.789	1.818
	1.447	1.843	1.778	1.793

^a Duplicates not reported.

RECOMMENDATIONS.

It is recommended that—

- (1) The modified J. L. Smith method for total potassium be further tested.
- (2) The sodium peroxid fusion method for total phosphorus be adopted as a provisional method of this association and be further tested.
- (3) The "Determination of Volatile Matter" (U. S. Dept. Agr., Bureau of Chemistry, Bul. 48, Revised, p. 72; Bul. 107, p. 14) be replaced by the "Determination of Total Organic Carbon" (J. Amer. Chem. Soc., 1904, 26: 1640).

REPORT ON INORGANIC PLANT CONSTITUENTS.

By W. W. SKINNER, *Referee.*

The work upon inorganic plant constituents has been a continuation of the scheme outlined for the preceding year. The chemists of six experiment stations signified their willingness to undertake the cooperative work, and samples were sent to each, but only a partial report from one station has been received. The lack of interest shown in the work has been rather discouraging to the referee, and it would seem that the objects of the association are defeated unless more cooperative work on proposed methods can be obtained.

The table given below is a tabulated statement of the sulphur content of two samples of cotton-seed meal and two samples of mustard-seed meal determined by three methods. The results are expressed on a moisture-free basis, and the sulphur calculated to sulphanion (SO₄). For purposes of comparison the sulphur content of the samples used the previous year are included.

TABLE I.—*Determination of sulphur as SO₄ in moisture-free cottonseed meal.*

Analyst.	Sample A.					
	Method employed.					
	Combustion.			Peroxid.	Ordinary ashing.	
	Nonvol- atile.	Volatile.	Total.	Total.	Total.	Loss.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
W. W. Skinner.....	{ 0.02 .05	1.62 1.56	1.64 1.61	1.73 1.74	0.20 .17	
Average.....			1.63	1.74	.19	.89
C. E. Goodrich.....				{ 1.69 1.67	.16 .13	
Average.....				1.68	.15	.91

TABLE I.—*Determination of sulphur as SO₄ in moisture-free cottonseed meal—Continued.*

Analyst.	Sample C.					
	Method employed.					
	Combustion.			Peroxid.	Ordinary ashing.	
	Nonvol- atile.	Volatile.	Total.	Total.	Total.	Loss.
W. W. Skinner.....	<i>Per cent.</i> 0.17	<i>Per cent.</i> 1.57	<i>Per cent.</i> 1.74	<i>Per cent.</i> 1.62	<i>Per cent.</i> 0.20 .16	<i>Per cent.</i>
Average.....				1.62	.18	.89
E. I. Lichtl.....	.43	1.78	2.21	{ 1.66 1.64	.16 .16	
Average.....				1.65	.16	.90
O. M. Shedd.....				{ 1.51 1.58		
Average.....				1.55		
Geo. Roberts.....				{ 1.65 1.61		
Average.....				1.63		

TABLE II.—*Determination of sulphur as SO₄ in moisture-free mustardseed meal.*

Analyst.	Sample B.					
	Method employed.					
	Combustion.			Peroxid.	Ordinary ashing.	
	Nonvol- atile.	Volatile.	Total.	Total.	Total.	Loss.
W. W. Skinnner.....	<i>Per cent.</i> 0.13 .21	<i>Per cent.</i> 4.11 4.15	<i>Per cent.</i> 4.24 4.26	<i>Per cent.</i> 4.37 4.43	<i>Per cent.</i> 0.40 .33	<i>Per cent.</i>
Average.....			4.25	4.40	.37	.92
C. E. Goodrich.....				{ 4.33 4.34	.39 .30	
Average.....				4.34	.35	.92

Analyst.	Sample D.					
	Method employed.					
	Combustion.			Peroxid.	Ordinary ashing.	
	Nonvol- atile.	Volatile.	Total.	Total.	Total.	Loss.
W. W. Skinner.....	<i>Per cent.</i> 0.40	<i>Per cent.</i> 3.67	<i>Per cent.</i> 4.07	<i>Per cent.</i> 4.18	<i>Per cent.</i> 0.46	<i>Per cent.</i> 0.89
E. I. Lichtl.....				{ 4.56 4.42	.47 .50	
Average.....				4.49	.49	.89
O. M. Shedd.....				{ 4.44 4.57		
Average.....				4.51		

The results for total sulphur by the peroxid method are fairly satisfactory and the referee recommends that this method, which is now provisional, and which was recommended last year for adoption, be made an official method.

Two of the analysts reported considerable difficulty with the peroxid method, especially with mustard-seed meal. This was probably due to one of two causes. Either the charge was not thoroughly moistened before the addition of the sodium carbonate and sodium peroxid, or it was not allowed to stand a sufficient length of time after the addition of the peroxid for the preliminary oxidation to occur. If this oxidation is not completed before the charge is placed over the flame, almost invariably the contents will be blown from the crucible by the violent action when it does begin.

The results by the combustion method are interesting and significant. The results for total sulphur, calculated to sulphuric acid (SO_4) agree within 0.1 per cent with the results by the peroxid method, while the results upon nonvolatile sulphur by this method are in striking agreement with the sulphur obtained in the ash by the ordinary method of ashing.

The combustion method is probably too slow and the technique too intricate for it to become the routine method for ash analyses. As an exact method for determining separately the volatile and nonvolatile mineral constituents of organic materials, it is, however, of great importance, and is therefore recommended for provisional adoption. The percentage loss of sulphur by the ordinary ashing method is shown in the table. On sample A, cotton-seed meal, the loss found by two analysts was, respectively, 89 per cent and 91 per cent, and on sample D, 89 per cent and 90 per cent. On samples C and D, mustard-seed meal, the loss was 92 per cent and 92 per cent, and 89 per cent and 89 per cent by two analysts.

The loss of sulphur by volatilization is very important in the interpretation of those proximate analyses in which ash is one of the factors. Thus in the analysis of a cattle food the determination of ash, by the ordinary method of incineration in air, constitutes one of the separations, the percentage of moisture, ash, fat, protein, and crude fiber being added together and subtracted from 100 to obtain the percentage of nitrogen-free extract. It is evident, therefore, that in a product with a sulphur content similar to mustard-seed meal, in which the loss of sulphur is about 4 per cent, the nitrogen-free extract would be just so much too high, provided the sulphur was not accounted for in some other form. The matter is further complicated by the fact that the sulphur is partially accounted for in the protein, as the protein figure is obtained by multiplying the nitrogen content by a factor which includes sulphur in the formula for the protein molecule.

When the association decided at the meeting in 1901 to adopt the name "Inorganic plant constituents" to replace "ash," by inference, at least, the older methods for ash were abandoned. The referee believes this was a mistake which has resulted in considerable confusion and to which attention was specifically called in his report for last year. At present the official method is by ignition with calcium acetate, which in many cases is impracticable. For instance, in the analysis of a forage crop one of the determinations is ash, and it is frequently important to determine in the ash the mineral constituents. If, however, the mineral constituents in the material are determined by the calcium acetate method it is impossible to translate them in terms of the ash as determined by the official method for ash in feeds and feeding stuffs. The result has been that workers along this line have continued to use the methods for ash given in Bulletin 46, Revised, although now not official. It seems advisable, therefore, that this method of ashing by charring, extracting, etc., be

again inserted in our methods as an official method, and the referee recommends this.

There has never been an official method for the determination of iron and aluminum in ash. It is therefore recommended that the development of methods for this purpose be especially referred to the referee for next year.

RECOMMENDATIONS.

It is recommended that—

- (1) The peroxid method be adopted as the official method.
- (2) The combustion method be adopted as provisional.
- (3) The official method for ash given in Bulletin 46, page 77, be again adopted.
- (4) The development of methods for determining iron and aluminum in ash be referred to the succeeding referee.

THE PHOSPHORIC ACID OF THE SOIL.

By G. S. FRAPS.

It is the purpose of this paper to present briefly some of the more important results which have been obtained at the Texas experiment station in the study of the phosphoric acid of the soil. The work has been confined chiefly to a study of the phosphoric acid soluble in fifth-normal nitric acid, and in ammonia under the conditions of the humus extraction.

AMMONIA-SOLUBLE PHOSPHORIC ACID.

The solubility of the phosphoric acid of mineral phosphates in ammonia was first studied, and it was found that ferric phosphate, vivianite, and wavellite give up a large part of their phosphoric acid to ammonia, while the calcium phosphates do not. It thus appears probable that some of the phosphoric acid soluble with humus in ammonia is of inorganic origin.

Twelve soils were then treated with potassium phosphate and a comparison made of the phosphoric acid soluble with humus before and after the treatment. We found an increase in so-called humic phosphoric acid in some of the soils, which proves that the absorbed phosphoric acid is soluble in ammonia, and some of the phosphoric acid soluble with humus must be of mineral origin. One of these soils, which is a subsoil and contains practically no humus, was nearly as rich as some black Minnesota soils in ammonia-soluble phosphoric acid, showing 0.06 per cent before and 0.228 per cent after treatment.

The distribution of the phosphoric acid in the ammonia extract as prepared for the estimation of humus was also studied. About one-ninth of the phosphoric acid is precipitated with the clay and is probably held in mechanical suspension. About one-third is precipitated with the organic matter and is probably in combination with it. About four-ninths remains in solution and is probably of mineral origin. These figures represent average results from seven Minnesota soils rich in humus.

The phosphoric acid associated with the organic matter does not diffuse and is not increased when the organic matter is precipitated in the presence of phosphoric acid, nor decreased when the organic matter is dissolved in ammonia and reprecipitated. It is concluded that the phosphoric acid is in organic combination.

The organic phosphoric acid is present in soils in much smaller quantity than has generally been assumed, even in soils rich in humus. On account of the smallness of the quantity in the soil and the slowness of decomposition of the

humus, the organic phosphoric acid can not be considered as of great importance, except possibly in some soils rich in humus, where it may serve as a reserve store of phosphoric acid.

An increase in the phosphoric acid soluble in ammonia during processes of decay does not necessarily mean that phosphoric acid has entered into organic combination.

PHOSPHORIC ACID SOLUBLE IN FIFTH-NORMAL ACID.

Efforts were also made to ascertain what mineral phosphates are affected by the fifth-normal nitric acid. Two thousand cc of solvent and 0.2 gram of phosphoric acid mineral were brought together for five hours at 40° C. and phosphoric acid was determined in the filtrate. It was found that practically all of the phosphoric acid of iron, aluminum, and calcium phosphates, and the minerals phosphorite, vivianite, triplite, and apatite was dissolved. Wavellite, dufrenite, and variscite were only slightly affected. Experiments with other solvents gave similar results.

The conclusion drawn from this work is that the solvents can not differentiate phosphates of different agricultural value in the soil. To take an extreme case, equal quantities of phosphoric acid in acid phosphate and apatite would yield the same results with fifth-normal acid and other solvents, but when applied to the soil the crops produced would be decidedly different. We are therefore justified in comparing the phosphoric acid extracted by weak solvents only with soils which probably contain the same phosphates.

The next point considered was whether the phosphoric acid dissolved from the mineral phosphates was removed from solution by the soil; that is, if any fixation from the fifth-normal nitric acid occurred. This question was studied by ascertaining the amount of phosphoric acid dissolved, first, from the soil alone, and second, from the soil plus a known quantity of phosphate. It appeared that a number of soils have a high absorptive power for phosphoric acid from solution in fifth-normal nitric acid. There were at least three soils in which over 70 per cent of the added phosphoric acid (210 parts per million) were absorbed. Similar results were observed with other solvents tested. It is evident that the phosphoric acid extracted from some soils may represent less than 30 per cent of the phosphoric acid which actually went into solution. Two soils with the same amount of phosphoric acid extracted by fifth-normal nitric acid may really contain quite different quantities of soluble phosphates.

This question of absorption must be seriously considered in connection with soil analysis. Its significance and the correction that should be made for it are being studied at the Texas Station. We are satisfied that the fifth-normal nitric acid method will yield results of great practical value. A large number of pot experiments have been conducted, but chemical analyses can now take the place of such investigations to some extent, and we expect to discontinue pot experiments upon certain types of soils containing less than 20 parts per million of phosphoric acid, as all of these soils appear to be seriously deficient in phosphoric acid. Full details of this work will be published by the Texas station at a later date.

REPORT OF COMMITTEE ON NOMINATIONS.

Mr. B. L. Hartwell, chairman of the committee on nominations, submitted the following report: For president, Mr. Harry Snyder, of Minnesota; vice-president, Mr. W. D. Bigelow, Washington, D. C.; secretary, Mr. H. W. Wiley, Washington, D. C.

For additional members of the executive committee, Mr. B. B. Ross, Alabama, and Mr. G. F. Fraps, Texas.

On motion by Mr. Frear, the secretary was instructed to cast a unanimous vote for the officers nominated.

On behalf of the committee on food standards, Mr. Frear submitted a report on the progress made and the work done jointly with the committee appointed by the National Association of State Dairy and Food Departments.

REPORT OF COMMITTEE ON FERTILIZER LEGISLATION.

During the last few years the committee on fertilizer legislation has considered the subject in various aspects. First: We have studied the problem of whether or not national legislation respecting interstate trade in fertilizers is desirable. Without question national legislation respecting the use of fertilizers in the District of Columbia is extremely desirable, and likewise in those Territories which have not legislated on the subject themselves. At the present there is no law whatever controlling commerce in fertilizers in the District of Columbia. This, however, as can well be seen, is not a matter of very great importance, because the tilling of the soil is not the principal industry of the District of Columbia. It is doubtful, therefore, if it would be advisable for Congress to legislate upon this subject solely for the District.

Second: This committee has also been in correspondence from time to time with the manufacturers of fertilizers. It is our opinion that national legislation would be highly beneficial to the manufacturers of fertilizers provided a system of tagging and a method of expression of analysis could be adopted which would be satisfactory to practically all the States, in which case one label would be legal throughout the country. Attention should be called in this connection to the effect of the national food and drugs act in bringing into harmony the definitions of adulteration and misbranding in the various States. Already from 15 to 20 States have adopted the definitions of the food and drugs act in respect of adulteration and misbranding, either by direct legislative enactment or by the establishment of regulations by the food and drug authorities. It appears quite probable that this is only a beginning and that it is only a question of a few years when the definitions of adulteration and misbranding of foods and drugs will be practically the same in all the States. If this happy conclusion could be reached, it would be sufficient reason for actively favoring the passage of a national law controlling interstate commerce in fertilizers and fertilizing materials.

It is evident, however, that there would be many points of dispute, not only between the officials of the various States respecting the proper method of tagging and of expressing analytical results, but also a still wider disagreement between the State officials and the manufacturers. It seems, therefore, unwise to press the subject of national legislation in regard to fertilizers further until the officials of this association, representing as they do the majority of States exercising fertilizer control, can come to an agreement respecting a uniform system of labels and of methods of expressing analysis. This subject, as is well known, has been under discussion by our association for several years, and satisfactory and encouraging progress has been made. This leads to the hope that in a few years more the representatives of each State in this association may come to an agreement on this important subject.

Third: The committee does not deem it wise to favor any form of national legislation which would in any way interfere with the State system of control

of fertilizers. That is a matter with which, in our opinion, the National Government has nothing whatever to do. The systems of control, as is well known, are not uniform. In some States a tax is laid upon the gross tonnage of fertilizers sold. In other States a tax is laid upon the labels which are attached to the various packages, and other forms of control are exercised. It is believed that the wisest control of this kind is that which seems best to the State officials who have charge of such matters, and the State legislatures which establish the legal status of such control. This committee, therefore, is opposed to any national legislation which would attempt to influence the States in any way respecting the method of control of fertilizer sales within the States.

Fourth: After repeated attempts to secure suggestions from manufacturers, your committee is of the opinion that upon the whole the manufacturers themselves would rather not have national legislation and prefer to submit to the disadvantages of the present system rather than to see incorporated in a national law any system of stating analyses or the character of the materials used in the compounding of fertilizers which would be objectionable to them. This, however, does not seem a sufficient ground upon which to advise that no national action be taken, and it seems advisable to endeavor to secure from the manufacturers a full expression of their views in order that the matter may be widely discussed. If it were possible to extend the agreement among the State officials already referred to relating to methods of tagging and of stating the results of analytical work, so as to obtain the approval of the manufacturers, the difficulties in the way of a national law would be practically eliminated. In this case the enactment of such a law would prove beneficial to all parties by aiding in securing the agreement among the various States.

Fifth: Your committee therefore recommends that for the present no attempt be made to bring a national fertilizer law to the attention of the Congress with the object of controlling commerce in fertilizers in the District of Columbia and the Territories of the United States and in interstate commerce, but that, on the other hand, an effort be made to secure an agreement among all the State officials respecting the fundamental definitions of the misbranding and adulteration of a fertilizer, and a common understanding respecting the proper method of tagging or branding, and the proper method of stating the results of analysis; that an attempt be made to secure an agreement between the officials of the States and the fertilizer manufacturers respecting the proper method of referring to the crude sources of the plant food which may be present in any given fertilizer. This committee believes that all these points should be settled before any concerted effort is made to bring the matter of national fertilizer control to the attention of Congress. Further than this, your committee is of the opinion that when such an effort is made it should relate solely to the fundamental conditions above mentioned, and should be so conducted as not in any way to affect the States in respect to the proper methods of raising revenue from fertilizers sold under the State control.

Sixth: The committee is aware that this report is not fully in harmony with the last recommendation of the committee published on page 175 of Bulletin No. 105 of the Bureau of Chemistry. It is there recommended that the committee be continued, with instructions to secure, if possible, collaboration of the great fertilizing interests in the perfection of a measure which in the future may be submitted to Congress with the joint approval of the manufacturing interests and of the Association of Official Agricultural Chemists. It is hoped that such a measure may be perfected, but precedent to that it seems wise that the points mentioned above should be first considered.

Seventh: It is therefore recommended that the committee be continued with instructions to confer with any committee appointed by this association relating

to the methods of branding or tagging and the methods of expressing analysis, and that when agreement is secured in this association on these points the great fertilizing interests be again approached with a view to securing their adhesion to these points, and also to the definitions of adulteration and misbranding which may be adopted by this association. As a tentative basis of discussion, the accompanying definitions of misbranding and adulteration are submitted.

H. W. WILEY.
B. W. KILGORE.
H. B. McDONNELL.
B. B. ROSS.
J. L. HILLS.^a

TENTATIVE DEFINITIONS OF FERTILIZERS AND OF MISBRANDING AND ADULTERATION.

(1) A fertilizer shall be defined as any simple, compound, or mixed material, prepared for the purpose of selling, or sold, or offered for sale, to be applied to the soil as nourishment for plants, or as a modifier of the soil in any respect in its relation to the growth of plants. The term "fertilizer material" (or ingredients) shall include every plant-food material which is utilized, or intended to be utilized, in the manufacture, preparation, or mixing of the fertilizers defined above.

(2) A fertilizer, or fertilizer material (or ingredient), shall be deemed to be adulterated:

(a) If the percentage of any of its ingredients fall materially below the professed standard under which it is sold, whether this standard appear as a label upon the package or as a guaranty in any other way by the vendor thereof.

(b) If any of the ingredients thereof have an origin other than that indicated upon the package, or guaranteed in any other way by the vendor thereof.

(c) If any of the ingredients of the fertilizer, or fertilizer material, be in a state of combination different from that indicated by the label or guaranteed by the vendor thereof.

(3) A fertilizer, or fertilizer material, shall be deemed as misbranded:

(a) If any false name or misleading statement or design or device be affixed to any package thereof or used in any way as a representation of the materials thereof by the vendor.

(b) If any false or misleading statement respecting the origin of the material be made upon the label, or any statement or guaranty of the vendor.

(c) If any false or misleading statement be made upon the label, or by the vendor, respecting the country or origin of the materials of which the fertilizer is composed.

(d) If any false or misleading statement be made on the label, or by the vendor, respecting the virtues or qualities of the fertilizer or the materials composing it.

^a Mr. Hills, in signing the report, submitted certain criticisms and suggestions bearing on the tentative definitions, calling attention especially to the following points: (1) The danger of injustice to a product arising from imperfect mixing and mechanical separation in transit in the application of a percentage standard, as indicated in "2 (a)." (2) The difficulty of interpreting under "3 (a)" the words "misleading statements" or "design or device;" for example, in the application of the term "guano," or the labeling of fertilizers stated to be especially adapted for certain purposes, as, for example, "potato fertilizers," found to be low in potash, and therefore not especially applicable for this purpose.

(e) If sold under any false name or appellation, whether such name appear upon the package or label or be given to the article by the vendor thereof.

(f) If it be an imitation of or offered for sale under the name of another fertilizer or fertilizer material.

The report of the committee was adopted.

REPORT OF COMMITTEE ON THE TESTING OF CHEMICAL REAGENTS.

By L. F. KEBLER, *Chairman*.

During the past year little of importance has appeared in print dealing with chemical reagents. E. Merck's book on "Chemical Reagents, their Purity and Tests," which has been translated into English by Henry Schenck, contains several new features which are interesting, but a number of new titles for chemical reagents have been employed which can only lead to confusion.

The committee during the past year has been unable to devote any time to cooperative work on chemical reagents. The chairman has, however, partially completed an investigation of the quality and purity of the indicators now supplied to chemists, and thus far the results indicate that these materials are far from satisfactory. One indicator in particular, namely, pure litmus cubes, should be mentioned. Most of us have been under the impression that these cubes represented a highly purified litmus product, when as a matter of fact they consist of calcium carbonate pressed into cubes and impregnated with a solution of litmus, from 94 per cent to 96 per cent of calcium carbonate being present. It is the intention of the chairman to make a full report on indicators at the next meeting.

A large number of chemicals have been examined during the past year in the drug laboratory, with the result that there is a general improvement in the quality of the reagents supplied to the Bureau of Chemistry, but it frequently happens that chemicals sold as possessing the highest degree of purity are found to be unsatisfactory, being contaminated with foreign material which renders them unsatisfactory for analytical work. These data are being collected and will be prepared for publication.

It is recommended that the committee be continued for another year.

REPORT OF COMMITTEE ON UNIFICATION OF TERMS FOR REPORTING ANALYTICAL RESULTS.

R. J. DAVIDSON, *Chairman*.

This committee has endeavored for nearly three years to get an expression of opinion from the members of the association as to their views in regard to the nomenclature for reporting analyses of fertilizers, etc., but only comparatively few have responded. The committee decided at its last meeting that it would be best to make a report at once without waiting for further replies to the circulars of inquiry sent out to the members of the association. The recommendations of the committee are as follows:

(1) That the nomenclature now in use for fertilizers, soils, ash, etc., be retained.

Adopted.

(2) That the association vote upon the advisability of permitting the use of a dual system of nomenclature, when desirable, with a view to the ultimate adoption of the element system for reporting the analysis of fertilizers, soils, ash, etc.

After considerable discussion Mr. Davidson called attention to the fact that this motion merely called for a vote for the consideration of the subject and did not commit the association to the support of the dual system of nomenclature. The motion was put and the second recommendation carried.

Following the adoption of these motions Mr. Hopkins spoke at some length in regard to the difficulties inherent in the present system of nomenclature, and read a number of letters received favoring the element system. An abstract of Mr. Hopkins's remarks is as follows:

MR. HOPKINS. In presenting some of the arguments in favor of the element system of nomenclature for soils and fertilizers, I may first mention briefly the action already taken.

In 1904, at the St. Louis meeting of the Association of Official Agricultural Chemists, a committee was appointed on unification of terms used in reporting analyses; and at the Des Moines meeting a similar committee was appointed by the Association of American Agricultural Colleges and Experiment Stations. These two committees have cooperated in the work assigned and at the annual meetings in 1906 made substantially the same report to their respective associations.

Final action on the report was postponed for one year by the Association of Official Agricultural Chemists at the Washington meeting in 1906, in part because at that time no action had been taken by the broader Association of Agricultural Colleges and Experiment Stations, whose annual convention was held a week later at Baton Rouge, at which meeting, however, the report, which includes the following statements, was adopted with the conditions stated:

The subject-matter referred to your committee naturally divides itself into two classes, one of which includes soils, fertilizers, ash, and other materials whose analysis may be expressed in terms of chemical elements or in simple compounds; while the other class includes foodstuffs, condiments, and other materials whose analysis may best be expressed in terms of more complex compounds (or groups of compounds) which actually compose or are contained in the material.

SOILS, FERTILIZERS, ETC.

Providing concurrent action is taken by the Association of Official Agricultural Chemists and the American Chemical Society, your committee favors the adoption of the element system for reporting analytical results in the analysis of soils, ashes, and fertilizers, as rapidly as possible, and recommends that the association urge those responsible for fertilizer legislation to have the laws changed, if necessary, and as soon as practicable, to meet with these recommendations, if concurred in.

This would necessitate the adoption of the dual system of expressing results temporarily in some States, but it is hoped that when fertilizer laws are adopted to meet these requirements some definite time will be set, at the expiration of which only the element system will appear on the bags or tags. It is therefore suggested that no other terms than those of the element system be allowed after the year 1916.

Your committee also recommends, provided the foregoing is adopted, that this association adopt some definite form for stating the composition of fertilizers and fertilizer materials.

We recommend that the terms "available" and "inert" shall be used in harmony with the construction placed upon them by the Association of Official Agricultural Chemists.

The committee also recommends that in case of the adoption of the foregoing there be required to be printed on the bag or on the tag to be attached to the bag or to accompany fertilizers sold in bulk an explanatory statement naming the materials in which the plant food is carried.

This report, with the conditions stated as presented by the committee, was adopted by the Association of American Agricultural Colleges and Experiment Stations at the Baton Rouge meeting.

You will all agree that it is desirable, where possible and practicable, to report in analytical statements constituents which are actually present in the material analyzed and which are of direct interest to the party for whom the analysis is made.

In fertilizers, such as dried blood, sodium nitrate, ammonium sulphate, or calcium cyanamid, the only constituent which is actually present in each material and which interests the users of fertilizers is the element nitrogen, and in terms of nitrogen a comparison can be made of these materials which would not be easily possible if the analytical statements showed nitrogen (N) in dried blood, ammonia (NH_3) or ammonium (NH_4) in ammonium sulphate, nitrogen pentoxid (N_2O_5) or the acid radicle (NO_3) in sodium nitrate, and possibly some other radicle or compound for calcium cyanamid (CaCN_2). There exists at present confusion in the matter of reporting nitrogen, although the tendency is clearly toward the use of the element system in this case. Under the fertilizer laws of several States nitrogen from any source must be reported in terms of ammonia (NH_3), and this is the most common method of statement in the wholesale markets involving organic nitrogen, but to report ammonia in sodium nitrate, or nitrogen pentoxid in ammonium sulphate, would be extremely confusing to say the least. The only rational and scientific basis providing uniformity is to report nitrogen in all of these materials in terms of the element.

In the case of potassium the same argument holds with equal force. Potassium is already being reported in terms of the element to a considerable extent, and the practice is increasing and probably will increase as rapidly as was the case in changing from ammonia to the element nitrogen. (See, for example, Bulletin No. 22 and others, of the Bureau of Soils; Bulletin No. 60 of the New Mexico Agricultural Experiment Station; Bulletins 182, 183, and others, of the Ohio Agricultural Experiment Station, and the experiment station bulletins and State fertilizer laws of Kansas and Illinois.)

The element phosphorus is most commonly reported in terms of the pentoxid (P_2O_5), which with chemical inaccuracy is termed "phosphoric acid." In most of the publications from the Bureau of Soils,^a and in the publication referred to from the New Mexico experiment station, phosphorus is reported in terms of the acid radicle (PO_4), while in the experiment station bulletins of Ohio, Kansas, and Illinois, it is reported as the element (P), and this is already

^a The following note by the chief of the Bureau of Soils is submitted in this connection to make clear the position of that Bureau in regard to the use of the element system:

* * * There seems to be a serious misapprehension on the part of several members of the association as to the position of the Bureau of Soils regarding the statement of analytical results. The Bureau of Soils follows the recognized practice of the U. S. Geological Survey and the foremost laboratories of this country and abroad. In stating the analytical results of analyses of water solutions the "ion" system, or the system founded on the hypothesis of electrolytic dissociation in aqueous solution, is employed. In other statements of mineral analyses the conventional "oxid form" is employed. In some few cases the "ion" system appears to conform to the proposed "element" system, but the conformity is by no means real. The "element" system has never been employed by the Bureau of Soils, nor has it ever seemed necessary or desirable to express an official opinion concerning it. * Digitized by Google

required by the fertilizer laws of two States. Phosphorus in fertilizers is also very commonly reported in terms of tricalcium phosphate, ordinarily known as "bone phosphate" or "lime phosphate" or "bone phosphate of lime." In many cases this is objectionable because the compound does not exist in the fertilizer, as in acid phosphate, dissolved bone, or basic slag.

In the matter of soil analysis almost insurmountable difficulties are met with in attempting to report the elements present in terms of compounds on a uniform comparable basis.

Berthelot and André^a report having found sulphur in the soil in the form of mineral sulphates, ethereal sulphates, metallic sulphids, and in organic nitrogen compounds of sulphur. They also report that more than one-half of the total sulphur of the soil is sometimes lost in ash determinations, and yet in most published reports of soil analysis sulphur is reported in the form of SO_2 whether contained in the soil in the form of sulphids or organic sulphur or as mineral sulphates.

In a recently issued text-book on soils, in four consecutive statements of soil analyses iron is reported as "ferric oxid," as "iron sesquioxid," as "iron protoxid," as "peroxid of iron," and as "oxid of iron." In volume 1 of the *Cyclopedia of American Agriculture*, published in 1907, manganese is reported as "manganese oxid (MnO)," as "manganese (Mn_2O_3)," as "manganese oxid (Mn_2O_3)," and as "manganic oxid (MnO)." In the same publication are soil analyses in which the sulphur is reported as "sulphuric acid," as "sulphuric anhydrid," as "sulphurous acid," and as "sulphuric trioxid."

There is but one simple basis for reporting an ordinary soil analysis and that is the basis of the chemical elements present. If necessary, we can report ferrous iron and ferric iron, which will give strictly comparable data, whereas the equivalent weights of ferrous oxid and ferric oxid are not directly comparable. Likewise we can report organic phosphorus and mineral phosphorus; sulphur in sulphids, in sulphates, and organic sulphur; also organic nitrogen, ammonia nitrogen, and nitrate nitrogen, and in all cases comparisons are direct and plain. To report the equivalent weights of NH_3 and NO_3 in ammonia salts and nitrate salts, because those radicals happen to be cations or anions, renders direct comparison impossible and serves no purpose of value to agriculture.

Think of reporting to the farmer that his soil is acid and in need of lime—perhaps 5 tons to the acre—and at the same time sending him an analysis showing that his soil contains 1 per cent of calcium oxid from which he finds by easy computation that his soil already contains 18 tons of lime per acre-foot. Then explain to him that the analytical statement is not correct, that his soil contains no calcium oxid, but that it contains sufficient calcium to make 1 per cent of calcium oxid if it were to be converted into calcium oxid, but into which it will not be converted, and that consequently his soil remains acid and does need lime.

Think of reporting an analysis of potassium chlorid as 63 per cent of potassium oxid and 48 per cent of chlorin (less oxygen misplaced, 11 per cent), and yet we have been doing just such unnecessary and unreasonable things in agricultural chemistry; meanwhile the farmers and farmers' institutes and the agricultural press continue to call upon science for the plainest, simplest statement of facts with the least possible use of unnecessary technical terms or expressions.

^a Ann. chim. phys., 1888, 15 [6]: 119.

In the chemistry of iron and steel when we deal with phosphorus we say phosphorus and we mean phosphorus, and in pharmacy and medicine when we say phosphoric acid we mean phosphoric acid, but in agricultural chemistry when we deal with phosphorus, organic or inorganic, we say and write phosphoric acid and we mean usually phosphorus pentoxid or sometimes the radicle of phosphoric acid (PO_3), but never true phosphoric acid unless we speak as the farmer's druggist, physician, or veterinarian, and then it should be understood that we mean phosphoric acid.

If we desire to report the oxygen in a soil, we can report the total amount by computation and by difference just as accurately as we do under the old system. As a matter of fact, few of the elements in soil analysis are weighed as oxids, most of them being determined either by volumetric processes or by the gravimetric determination of some salt, as, for example, barium sulphate, potassium platinic chlorid, magnesium pyrophosphate, etc.

If the interest in any constituent concerns the compound present, then, in addition to the statement of elements, there should be given the percentage or amount of such compound. Thus the calcium carbonate present in the soil may be reported as such, not because of any element of plant food which it contains, but because of its value as limestone. Indeed, this is already being done in cases where the importance of calcium carbonate, as such, is recognized.^a

The only valid objection to the adoption of the element system for reporting analyses of soils and fertilizers is that most analyses have been previously reported on the basis of oxids, and consequently there would be some additional burdens, temporarily, in making comparisons with earlier work until the more important existing data shall have been compiled and recalculated to the element basis. This objection, which will apply to a few people for a few years, is extremely slight compared with the advantages of the simpler system to all people for all time; and it is worth while to call to mind in this connection that our forefathers discarded "pounds, shillings, and pence" for the decimal system, with its dollars and cents, at a time when the change concerned not a small proportion but practically the entire population of the country; also, that nearly all other countries, except Great Britain, have subsequently adopted a decimal money system; and, not only that, but they have also adopted decimal systems of weights and measures, a change involving temporary inconvenience and expense for almost every business in the country.

International agreement in the adoption of the element system for soils and fertilizers is desirable; but, even though the element system should never be adopted in European countries, it is nevertheless highly desirable that it should be adopted in this new agricultural country and that American agriculture should have the benefit of using the simplest basis in the application of science to this fundamental industry, upon whose ultimate success practically all other great industries depend.

The advantages of the simple element system for soils, fertilizers, and plant-food elements in farm produce are so great that no State having once adopted the element nitrogen in place of ammonia has ever returned to the ammonia basis, and for the benefit of her own agriculture any single State can adopt with advantage and profit the more complete element system, even though other adjoining States may retain some other system.

The association adjourned until 2 o'clock.

^a J. Agri. Sci., December, 1907, 2: 306.

FRIDAY—AFTERNOON SESSION.

The discussion of the adoption of a dual system of nomenclature looking to the adoption of the element system for reporting analyses of fertilizers, soils, etc., was resumed, and Mr. Hopkins offered the following motion:

That the suggestion of the committee looking toward the ultimate adoption of the element system be approved, but that no State should discontinue the use of the terms now in use until such discontinuation is also approved by this association and that meanwhile the subject should be brought before the International Congress of Applied Chemistry in an effort to secure international agreement;

That this association appoint a committee to bring the matter of reporting results according to the element system before the next International Congress of Applied Chemistry with power to add to their numbers at their discretion.

A lengthy discussion followed the seconding of this motion, during which the following opinions were expressed:

H. W. WILEY. I favor referring the matter to the International Congress of Applied Chemistry. It must be remembered that the elements are not present as such in the fertilizers, and the labels should show the definite composition of the package. Ethically this is always the requirement for a true label, but a statement might be made that the materials present equal so much phosphorus or nitrogen.

EDWARD GUDEMAN. It seems to me that a distinction should be made between the reporting of fertilizers and soils. Commercial conditions and common practice should be considered, and I can not see that the farmer will be benefited or obtain any more information from the element system than from the present one. The Council of the American Chemical Society is to consider this question at the December meeting and I shall oppose the adoption of the element nomenclature.

B. W. KILGORE. It seems to me that before going to the International Congress we should decide whether or not as an association we indorse the element system, and the motion as submitted does not seem to simplify the matter sufficiently. I can not see that the association comes any nearer a solution of the problem by sending such a report to the International Congress.

B. L. HARTWELL. Would it not be well to adopt the element system provisionally upon its adoption abroad and to send a committee to represent the association, approving such action if it can be made international?

H. J. WHEELER. I am in favor of the association calling the matter up and of the adopting of the element system if it can be made uniform usage under the State fertilizer law and by international agreement. In other words, I am opposed to the adoption of the element system under present conditions, but would approve it if universally adopted.

C. G. HOPKINS. It is not expected that any State would discontinue the use of its present system until further action is taken.

G. S. FRAPS. I am not willing to make this conditional indorsement of the element system. I believe that the association should decide this matter for itself and not pass it on to some other body for decision. The States accustomed to the old system will not be willing to begin their educational work over again in order to introduce a system which, to say the least, does not offer any advantage over that in use. We have uniformity practically all over the world in this

matter of expressing analyses of soils and fertilizers, with the exception of the States of Illinois, Ohio, and Kansas. It would be easier for these few States to abandon their new methods than for the remainder of the world to change methods of nomenclature which have been in use ever since the analysis of soils and fertilizers was begun. I endeavor to stand on the side of progress, and have honestly tried to see in what the merit of the elementary system of nomenclature consists, but it appears to me that its simplicity is a matter of words rather than facts, and that the old system is entirely preferable.

J. T. WILLARD. Kansas has adopted the element system without waiting for further action by the association. It makes no difference to the chemist or to the fertilizer manufacturer in respect to calculations how the results are stated. The farmers will require instruction in any case and can learn one system as well as another. Those who have learned the old system would be inconvenienced temporarily in learning the new. In the States where fertilizers have just come into use it would appear to be simpler to use the same terms in instructing the farmers, through bulletins and at institutes, as are used in instructing their children who study chemistry in our high schools and colleges. My plea is for the use of the right name on the label even at some temporary inconvenience, and not to saddle future generations with the present antiquated system. Had our forefathers been willing to endure a little difficulty in the introduction of the metric system we and our children would not be burdened with our complex and confusing tables of weights and measures.

B. W. KILGORE. The students in our agricultural colleges must understand the terms as at present used, and if they are to make any use of the literature on the subject they must deal in these terms.

B. B. ROSS. In response to a circular letter with reference to this subject which was sent out several years ago I expressed an opinion favorable to the use of the element system in reporting the results of analysis of soils and ash, but so far as its employment in reporting fertilizer analyses is concerned, I think it would be very unwise to take such a step for some time to come, and I do not think we should go on record as an association in favor of abandoning the present method of reporting fertilizer results.

Alabama was the first State to adopt the use of the term "nitrogen" instead of "ammonia" (NH_3), and we still find it necessary to explain the relation between the two. The authorities in our State connected with the fertilizer control have had great difficulty in securing the enactment of fertilizer legislation which is practically uniform with that of four or five adjacent States, and hence they are opposed to any change in the present system of reporting fertilizer analyses. For these reasons I must vote in opposition to the adoption of the element system for reporting results of fertilizer analyses.

J. G. LIPMAN. Many laws become obsolete when they lose the moral support of the people for whom they are framed. In this, as in other matters, the line of least resistance is always followed. Rules laid down for the guidance of men of science may become obsolete for the same reason. Now, there is evidently a strong sentiment in favor of the element system among some of the members of the association, and pronounced opposition to it among others. In adopting a dual system we give the element system a provisional approval. It can be discontinued when found inexpedient.

Only those chemists engaged in official fertilizer control work having a right to vote according to article 2 of the constitution, a rising vote was taken and the motion carried by a vote of 23 to 14.

The committee on the President's address, 1906, offered its report, which was accepted by the association.

REPORT OF COMMITTEE ON RESOLUTIONS.

Your committee submits the following report and recommends the adoption of the resolutions presented:

(1) Within the past few weeks Dr. George C. Caldwell, of Cornell University, and Dr. W. O. Atwater, of Wesleyan University, have been removed by death. Both these men were pioneers in agricultural chemical teaching and investigation in America, and as such have exerted a deep, abiding influence upon the progress of agricultural chemistry not only by their personal work but widely through the many who as students have received inspiration from personal contact with these men. Doctor Caldwell was an active member of our association as long as his health permitted, serving in the presidency one term and several times acting as referee. While Doctor Atwater was not directly active in our work, he was always in full sympathy with it. Many of us feel a distinct sense of personal loss in their death.

Be it therefore resolved, That we, as an association, express our sorrow at the removal of Doctor Caldwell and Doctor Atwater from earthly activities, and also our profound appreciation of the fundamental work which they performed as pioneers in agricultural chemistry.

Be it further resolved, That the secretary be requested to prepare for insertion in our proceedings appropriate minutes regarding the work of Doctor Caldwell and Doctor Atwater. (This resolution was adopted by a rising vote.)

(2) Whereas it is the belief of this association that great good may be accomplished for the unification and improvement both of analytical methods and of definitions of standards for the foods and drugs that enter into international commerce, by an international conference of chemists and others connected with food and drug control and with the administration of laws relating thereto;

Therefore, be it resolved, That this association favors such an international conference and requests the proper officials of the United States Government to take the initiative therein. [Resolution adopted.]

(3) *Resolved*, That the changes suggested by President Street in his address be adopted as follows:

That one day of our convention be devoted entirely to the reading of papers and their discussion, and that for this purpose the convention be divided into three sections covering the same subjects as our present committees on recommendations, and that all papers to be read in these sectional meetings be referred to the proper committee, with full power to reject the same or assign places to them in the programme sent to all members prior to the convention. [Resolution adopted.]

Signed: L. L. VAN SLYKE,
 CYRIL G. HOPKINS,
 W. A. WITHERS,
 Committee on Resolutions.

REPORT OF COMMITTEE ON AMENDMENTS TO THE CONSTITUTION.

(1) Your committee respectfully recommends that Article IV of the constitution shall be amended by the insertion in the second line, after the word "meeting," of the clause "from among the members of the association."

[This recommendation was adopted, and in accordance with the suggested change Article IV of the constitution reads as follows:

There shall be appointed by the executive committee at the regular annual meeting, *from among the members of the association*, a referee and such associate referees for each of the subjects to be considered by the association as that committee may deem appropriate.]

(2) With respect to the suggested change in the tenure of office and organization of the committees on recommendations, your committee believes that, for the present, the matter could be better disposed of by the adoption of a resolution than by the insertion of a new article in the constitution.

It recommends for the attainment of the chief purposes of the proposed changes, the adoption of the following resolution with its preamble:

Whereas it is desirable to secure the greatest practicable uniformity in the form of expression and arrangement of the methods adopted by this association, and also to provide for the thorough consideration of the various changes in methods and of new methods proposed;

Resolved, That, beginning with 1908, the committee on recommendations be composed of nine members, elected (*appointed by the president*) as follows: Three to serve for six years, three for four years, and three for two years; and that as their respective terms expire, the vacancies caused thereby be filled by the election (*appointment*) of qualified successors for the full term of six years.

Resolved further, That this committee shall organize from its membership three subcommittees, corresponding to the present committees A, B, and C, to whom all recommendations of new methods or of changes in methods, provisional or official, shall be submitted not later than three weeks prior to the date of the meeting of the association at which it is desired that the recommendations in question shall be considered, and said committee on recommendations shall report to the association, with its approval, disapproval, or suggestion of amendment all recommendations submitted in due form for its consideration.

[The difficulty of electing such a committee by the association as a whole and insuring that the members be well chosen, and also the method of filling vacancies occurring during the year, were discussed at some length and a motion to amend this resolution to read "appointed by the president," instead of "elected," was passed. The original resolution as amended was then adopted.]

(3) With respect to the suggested expansion of the territorial extent of this association, your committee most cordially seconds the appreciation of good fellowship and of mutual scientific advantages gained by the attendance of our Canadian confrères in this and earlier years, and further is convinced that many advantages would result from the harmonizing of the official control methods and standards used by our North American neighbors and ourselves. The changes suggested are, however, far-reaching in their effects and may change the status of the association in ways not intended. It is believed that before action the subject should receive full consideration.

We therefore recommend that the following amendments to the constitution be brought to the notice of the association in the published announcement of the next annual meeting for the action of that meeting:

In Article I, first sentence, substitute for the words "the United States" the words "North America."

In Article II, first sentence, second and third lines, insert the word "provincial" after the word "State;" also in the third line of this sentence insert after the word "body" the phrase "in North America."

[This recommendation was adopted, and the proposed amendment is placed on record for action at the 1908 meeting.]

WM. FREAR, *Chairman*.

B. W. KILGORE.

J. G. LIPMAN.

Col. J. Rice Smith, then addressed the meeting and spoke most enthusiastically of the chemist's work in its relation to the fertilizer trade:

This is the body of men to whom we look for the information which it is so necessary and important for us to have in the proper conduct of our business, and you are of more value to us than the lawmakers in Congress. We are working along the same lines and for the same purposes—to develop and build up the industries of this country, and on our part especially the agricultural industry. Everything that we buy goes through the laboratory and everything that we sell does the same, that we may know what we are getting and what we are giving. Our chemist is at the top of the building—and that is where he belongs in relative importance. It is the laboratory first and last with us, and I do not flatter you, gentlemen, when I say this association is of more value to us than any other body of men. We have problems to meet, and only the chemist can solve them for us. In order that we may give every one the value of his money and have our material uniform and true to label, the chemist must furnish us with the facts and we can put the information to intelligent and practical use. The chemist is always showing us how to turn our waste material into wealth. Thirty or forty years ago cotton seed—then the burden and waste of our great cotton crop—were in the way or thrown away, while to-day, throughout the civilized world, millions of dollars' worth of meal, oil, and other products are made from this source and utilized for feeding, fertilizing, and domestic purposes. It was only through the science of chemistry and the work of the chemist that this result could have been accomplished. The possibilities lying dormant in this country, if we develop them, will enable us to clothe and feed the world, and it is to chemistry that we must look for the information that will enable us to do it. Any improvement that you can suggest regarding our industry we will be glad to act upon. I thank you for your courteous attention.

REPORT ON PHOSPHORIC ACID.

By B. W. KILGORE, *Referee*.

The amount of American data available for determining the fertilizing value of Thomas or basic slag in comparison with other materials furnishing phosphoric acid is not large, but the territory over which the material is used and the amount employed for fertilizing purposes are annually increasing. It is therefore desirable to adopt a tentative or provisional method for its analysis and fix a basis for stating its commercial value. Until further data are obtained in connection with experiments which are now in progress or which may be instituted in the future the following recommendations are made:

RECOMMENDATIONS.

It is recommended—

(1) That Thomas or basic slag be prepared for analysis as other fertilizer or fertilizing materials are under the methods of this association.

(2) That the solution for analysis be made according to a-7, Bulletin 107, page 2, or in strong hydrochloric acid alone. In the latter case, after the portion for analysis is measured out, add nitric acid and heat for a few minutes. Then determine total phosphoric acid according to the official methods.

(3) That the fineness of the material be determined according to the plan followed with bone meal and the commercial value estimated on the basis of total phosphoric acid and fineness of product.

SUBREPORT ON PHOSPHORIC ACID.

By J. M. McCANDLESS, *Referee*.

On June 3, 1907, the following letter, together with one sample of Tennessee phosphate rock and two samples of acid phosphate, was sent to about twenty analysts, who had expressed a willingness to cooperate in the work:

DEAR SIR: I have sent you by express a box of samples for the cooperative work of the association on phosphoric acid and should be pleased to receive results on this work at your earliest convenience. To those who are interested in the question of the effect of sulphuric acid on iron pyrites in phosphate rock I would recommend that they add to a weighed portion of the rock 10 per cent of its weight of high-grade iron pyrites, then treat with sulphuric acid as described in (1) and determine whether any additional iron has passed into solution from the pyrites. This idea occurred to me too late to inclose a sample of the pyrites with the other samples.

METHODS TO BE USED IN THE DETERMINATION OF IRON AND ALUMINUM IN PHOSPHATE ROCK.

It is recommended that before beginning the work on a sample of phosphate rock a solution be made up imitating a phosphate rock solution and containing known quantities of iron and aluminum.

Make the solution about as follows:

Weigh 18.25 grams microcosmic salt; 0.400 gram iron wire; 8 grams recrystallized potash alum; 12.5 grams C. P. calcium carbonate; 0.300 gram calcium fluorid. Transfer to a liter flask, add dilute hydrochloric acid till carbonate is all in solution, and then more hydrochloric acid to dissolve the iron. Heat till solution is complete, cool, and make up to mark with distilled water.

It is earnestly recommended that the following methods be tried for your own satisfaction on this solution, in which you will know the percentage of iron and aluminum, before beginning work on the sample of phosphate rock.

I send only one sample of Tennessee phosphate rock, desiring to make the work as light as possible, and thinking it better to apply all the methods to one sample rather than to send a number of samples, which would involve much labor and probably result in less valuable work being done.

Inasmuch as in the manufacture of acid phosphate the rock is nearly always treated with about its own weight of 50° B. sulphuric acid, and as, where the rock contains clay, more Al_2O_3 is extracted by this treatment than is usually dissolved by hydrochloric acid in the methods of analysis prevailing, I have thought it wise to imitate the process of manufacture and to treat the rock in all the following methods with 50° acid before bringing it into solution with the hydrochloric acid.

Methods for alumina.

(1) *Thiosulphate method, modified.*—Treat 2.5 grams of the sample of phosphate rock (previously treated with a magnet to remove small particles of iron derived from the pulverizing implements) with an equal weight of sulphuric acid (sp. gr. 1.53) and heat on water bath for thirty minutes. Boil with 50 cc of 1:1 hydrochloric acid for thirty minutes and make up to 250 cc. Filter off 100 cc, equivalent to 1 gram, add about 100 cc of water, and bring to a boil. Remove from flame and add a slight excess of ammonia. Clear with hydrochloric acid, added drop by drop with constant stirring, then add 2 cc of concentrated hydrochloric acid in excess. Add 10 grams of sodium hyposulphite and 15 cc of acetic acid (sp. gr. 1.04) and boil for fifteen minutes. Filter on a rapid ashless filter paper and wash with hot 5 per cent ammonium nitrate solution. Ignite at a low temperature till paper is charred and then increase the heat gradually till carbon and sulphur are oxidized. Finally blast for one minute and weigh as phosphate of aluminum, containing 41.85 per cent of Al_2O_3 .

(2) *Gladding method.*—Treat 2.5 grams of the sample as above with an equal weight of sulphuric acid (sp. gr. 1.53). Heat with 50 cc dilute hydrochloric acid (1:1) and keep just below the boiling point for thirty minutes. Make up to 250 cc and filter off 100 cc (1 gram). Add a few drops of nitric acid to oxidize the iron and boil for a few minutes. Run this solution into 20 cc of a solution of C. P. caustic potash, made by dissolving 500 grams of caustic potash, free

from alumina, in distilled water and diluting to 1 liter. Digest in water bath at 70° for one hour, stirring occasionally. Let the precipitate settle, and filter on a large filter, first decanting the supernatant liquor on the paper, and finally transferring the precipitate to the filter. Wash the precipitate two or three times with hot water. To the filtrate add 1 gram of ammonium phosphate, acidify with hydrochloric acid, add ammonia till a permanent precipitate is formed, and then dilute hydrochloric acid drop by drop until it is just dissolved. Add a mixture of 15 cc of neutral ammonium acetate and 5 cc of acetic acid (30 per cent) and digest for half hour at 70°. Filter, washing five or six times with hot 10 per cent ammonium acetate solution, stirring up the precipitate each time with the jet from the wash bottle. Ignite with a low flame till the paper is charred. Increase the heat, and when the paper is entirely consumed blast for one minute. Weigh and calculate the percentage of Al_2O_3 as in (1).

(3) *Glaser method*.—Treat as in (1) up to the point when 100 cc, or 1 gram, has been filtered off. Add 25 cc of concentrated sulphuric acid, shake, and allow to stand a few minutes. Add about 100 cc of alcohol and cool. Make up to 250 cc with alcohol and allow to stand thirty minutes. Filter off 100 cc, equivalent to 0.4 gram. Evaporate to expel alcohol, using a rather large, hard glass beaker. Transfer to a smaller beaker and add a few drops of nitric acid to oxidize the last traces of alcohol and boil. Remove from the flame and cautiously add ammonia to slight alkalinity. Boil to neutrality; allow to settle and cool. Filter and wash with hot ammonium nitrate solution (10 per cent). Burn and weigh. Deduct from the weight the iron phosphate found volumetrically, to obtain the percentage of $AlPO_4$, or divide the weight found by 2 to obtain the per cent of the combined oxids of the iron and aluminum.

Oxid of iron.

Use any volumetric method you prefer for the determination of the iron.

The following method has been used by us and found to be very satisfactory:

Take 100 cc of the filtered solution used in the determination of the alumina under (1), transfer to a 500 cc flask, add a few crystals of potassium chlorate, and boil till chlorin has been removed. Add 3 grams of granulated zinc and 5 cc of hydrochloric acid. Place a funnel in the mouth of the flask and heat slightly till the iron has all been reduced and the zinc nearly all dissolved, then add 10 cc of sulphuric acid diluted with 20 cc of water, and boil till all the zinc has gone in solution. Make the solution up to about 500 cc with cold water and pour into 500 cc of cold water in a large white dish. Rinse the flask and add the washings to the dish. Titrate with standard potassium permanganate, making a correction for the iron in the zinc.

It is desired that you determine the percentage of insoluble phosphoric acid in the two samples of acid phosphate sent, using, first, the ammonium citrate as usually made in your laboratory; secondly, a solution of ammonium citrate made according to the following method:

Weigh out a suitable quantity of citric acid and calculate the approximate amount of ammonia that should be added to it to neutralize it. It has been our practice to weigh 7.5 pounds of citric acid, then pour on about 2 liters of water and add 3,100 cc of ammonia (6.90 sp. gr.) to it. Stir till all the acid has been dissolved and allow to cool. The solution gets very hot, and there is a loss of ammonia, leaving the solution acid. Make up to a definite volume, about 10 liters. Measure carefully 25 cc of this solution into a 250 cc flask, make up to the mark with distilled water, measure out 25 cc of this solution (corresponding to 2.5 cc of the original solution), and transfer to a distilling flask. Add an excess of standard potassium or sodium hydroxid and distil the ammonia into standard acid. Titrate and calculate the amount of ammonia, and when cool titrate the excess of caustic alkali in the residue in the distilling flask, using phenolphthalein as indicator, and calculate the amount of citric acid. Knowing the amount of ammonia and citric acid in the measured volume, calculate the quantity of ammonia necessary to neutralize the excess citric acid in the main body of the solution. Add this quantity from a solution of ammonia of known strength, shake and bring to a specific gravity of 1.00. This solution made neutral by analysis reacts alkaline both to litmus and to corallin. It also shows alkaline by the calcium chlorid test, but it will be found on trial that though most of the citric acid is removed from the ammonium citrate by alcoholic calcium chlorid, that a notable quantity still remains in solution, and that therefore this test is not so reliable as has been thought,

Unfortunately, pressure of other duties must have prevented the analysts from cooperating, as only the following results on iron and alumina had been received:

TABLE I.—*Determination of alumina and ferric oxid.*

(a) SYNTHETIC MIXTURE.

Analyst.	Sample.	Determination.	Method.	
			Gladding.	Thiosulphate.
Stillwell and Gladding (New York City).	Synthetic mixture.	Alumina (Al_2O_3)..	<i>Per cent.</i>	<i>Per cent.</i>
			12.42	12.23
McCandless and Burton (Georgia State Laboratory).	3 per cent Fe_2O_3 ; 5 per cent Al_2O_3 .	Ferric oxid (Fe_2O_3)..	12.44	12.48
		Alumina (Al_2O_3)..	3.03	3.05
			5.10	5.07

(b) TENNESSEE ROCK.

Analyst.	Alumina (Al_2O_3).			Ferric oxid (Fe_2O_3).
	Gladding method.	Thiosulphate. method.	Glaser method.	
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
McCandless and Burton (Georgia State Laboratory).	5.40	5.35	5.81	3.94
	5.32	5.40		
Stillwell and Gladding (New York City).....	6.44	4.73		
	6.74	4.95		
J. H. Mitchell (Clemson College, S. C.).....	5.27	5.38	5.33	
	5.27	5.48	5.28	4.54
F. B. Carpenter (Virginia-Carolina Chemical Company, Richmond, Va.).....	5.33	5.53	5.25	
		a 5.34		b 4.48

^a Average of seven determinations.

^b Obtained by calculating all the percentages of Al_2O_3 .

It is unfortunate that a greater number of chemists did not engage in the work, as the results of three of the chemists compare favorably on the percentage of alumina by the first two methods, but are offset by the wide difference in the results of the author of the Gladding method.

Subsequent experience by the referee has, however, shown that the thiosulphate method will, with certain rocks, give results that are far too low, and that it can not, therefore, be recommended in its present form. The simplicity and rapidity of the method, however, make it very desirable that the next referee continue work on it and endeavor to ascertain the cause of the low results obtained with certain phosphate rocks. At first the referee believed this to be due to the presence of fluorin, but the excellent results obtained in synthetic mixtures containing varying quantities of fluorids, discredit this view.

The referee added to three samples of finely ground rock 10 per cent of their weight of high-grade pyrites and then treated the mixture as outlined in Method (1). Careful determinations of iron in the resulting solutions showed not more than 0.1 per cent additional of ferric oxid to have been brought into solution, confirming the results of experiments made some years since by the referee and published in the American Fertilizer. Unfortunately, no other chemist to whom samples were sent reported any experiment along this line.

Two samples of acid phosphate, Nos. 2 and 3 in the following table, were sent out with the request that the insoluble phosphoric acid be determined, first, with ammonium citrate as usually made in the laboratory, and, secondly, by

the solution made as prescribed by the referee. Unfortunately, only two other chemists besides the referee and his associate have reported results on these two samples.

TABLE II.—*Determination of insoluble phosphoric acid.*

Analyst.	Ammonium citrate.		
	Slightly acid to litmus.	As ordinarily made with corallin.	Made neutral by analysis.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
	SAMPLE NO. 2.		
H. L. Davidson, Virginia Department of Agriculture.....	6.42	6.88	6.98
Robert H. Kerr, Lazaretto Guano Works.....		6.16	6.20
McCandless and Burton, Georgia Department of Agriculture.....	4.12	5.90	6.23
F. B. Carpenter,* Virginia-Carolina Chemical Company, Richmond, Va.....		7.00	7.12
		7.05	7.10
		7.05	6.99
	SAMPLE NO. 3.		
H. L. Davidson.....		4.08	4.11
Robt. H. Kerr.....		3.72	3.70
McCandless and Burton.....	4.15	4.23	4.22
F. B. Carpenter,* Virginia-Carolina Chemical Company, Richmond, Va.....		4.06	4.05
		3.98	4.00
		4.10	4.07

* Analysts, Henry and White.

It has been the experience of the referee that with the average acid phosphate slight differences in the neutrality of the ammonium citrate solution cause little or no variation in the percentage of insoluble phosphoric acid, but that occasionally samples of acid phosphate are found in which slight variations in neutrality cause very wide variations in the percentage of insoluble phosphoric acid. Sample No. 2 was of this character, and the referee therefore regrets that a greater number of chemists did not cooperate in the work.

A paper was submitted by Mr. Carpenter giving some figures obtained by procedures varying from those outlined by the referee, and the results of these studies are submitted as a supplementary report as follows:

SUPPLEMENTARY REPORT BY F. B. CARPENTER.

When sulphuric acid is used in the preliminary digestion according to your instructions, the results seem to be more uniform and satisfactory than when solution is made simply with hydrochloric acid. We tried making solutions with both concentrated and dilute hydrochloric acid, but it did not seem that it made any material difference in the results. Boiling one-half hour with concentrated hydrochloric acid without previous treatment with sulphuric acid does not seem to be sufficient time to decompose all of the iron and alumina phosphates in the sample under consideration.

The modified acetate method as used to obtain the results in the table is as follows:

Weigh 2 grams of sample in a 200 cc flask, add 30 cc of strong hydrochloric acid c. p., boil half an hour. Dilute, cool, make up to mark, mix, pour through dry filter. Measure 50 cc into an 8-ounce beaker. Oxidize by boiling with a little nitric acid, remove the beaker from the flame, neutralize with ammonia, and then just dissolve the precipitate in diluted hydrochloric acid, adding drop by drop, taking care to add only sufficient acid to dissolve precipitate.

Add 30 cc saturated solution ammonium chlorid, add water, filling the beaker to within about three-fourths of an inch from the top, place in water bath and heat to 70°. Remove from bath, add 5 or 6 drops of methyl orange solution, and then add ammonium acetate solution which has been heated to 70° until color changes from red to yellow. Add an excess. Filter on folded filter, and wash two or three times with hot 5 per cent solution of ammonium nitrate; dissolve this precipitate back into the same beaker, using diluted hydrochloric acid and hot water. Add 1 gram of ammonium phosphate to the solution and precipitate with ammonium acetate, proceeding as before. Filter and wash the last precipitate with 5 per cent solution of ammonium nitrate five times and once with hot water.

TABLE III.—*Determination of alumina by the thiosulphate method and modifications.*

[The totals are averages.]

Method described by reference, thiosulphate method No. 1.	Method No. 1 with addition of 3 per cent calcium fluo-rid to flask after weighing sample.	Modified acetate method, solution made as in method No. 1.	
<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	
5.45	5.08	5.21	
5.30	5.07	5.18	
5.25	5.20	5.15	
5.51	5.08	5.10	
5.30	5.17	5.16	
5.25	5.00		
5.34	5.10		
5.34	5.10	5.16	

Solution made by boiling 2 grams of rock one-half hour with concentrated hydrochloric acid without previous treatment with sulphuric acid.		Solution made by boiling 2 grams of rock one hour with 50 cc of concentrated hydrochloric acid.	
Thiosulphate method.	Modified acetate method.	Thiosulphate method.	Modified acetate method.
<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
4.91	4.47	5.00	4.90
4.85	4.45	5.15	4.77
4.73			
4.83			
4.83	4.46	5.08	4.84

We have not done sufficient work by the modified acetate method to vouch for its accuracy, but we believe that with some slight modifications it will be an ideal method. The advantage this has over the regular laboratory method, which we have previously used, is the elimination of any considerable amount of lime from the precipitate. In method No. 1, unless great care is taken in the manipulation, there will be some lime left in the precipitate and the results will be correspondingly high. In the hands of different chemists, especially those with little experience, our method has not always given good results, but after having had considerable experience with the method the analysts usually get excellent results.

PRELIMINARY STUDIES ON THE ANALYSIS OF BASIC SLAG FOR AVAILABLE PHOSPHORIC ACID.

By H. D. HASKINS.

The slag used for the following tests analyzed 19.02 per cent, 18.94 per cent, and 19.02 per cent total phosphoric acid, or an average of 18.99 per cent.

In view of the fact that it is generally conceded that the beneficial effect of Thomas slag phosphate on growing vegetation may be due, in part at least, to

the free lime present in the slag, it occurred to the writer that the fairest way of treating the slag for the determination of the available phosphoric acid was first to neutralize the free lime and finally treat the resulting product with neutral citrate of ammonia in the usual manner. This was accomplished by the two following methods:

Method A.—Two grams of the Thomas phosphate powder were weighed into an Erlenmeyer flask of 150–200 cc capacity, 10 cc of distilled water and a few drops of phenolphthalein solution as an indicator were added. The flask was then placed on a boiling water bath and when the solution became thoroughly heated, dilute citric acid was run into the flask until the pink color disappeared. After the mass had been brought to dryness, 100 cc of neutral citrate of ammonia were added and the digestion was carried on at 65° C. in the usual way as for the determination of citrate-soluble phosphoric acid. This method showed 6.61 per cent and 6.29 per cent of available phosphoric acid.

Method B.—Two grams of the Thomas phosphate powder were transferred to a small beaker, 10 cc of distilled water and a few drops of phenolphthalein solution were added and the beaker was placed on a boiling water bath. When hot, an excess of dilute citric acid solution was added and the mass was digested for fifteen minutes on the hot water bath; the solution was then filtered into a 200 cc flask and washed with hot water until it was up to the 200 cc mark. The residue was treated with neutral citrate of ammonia in the usual way and showed 6.83, 6.37, and 6.80 per cent of available phosphoric acid. The citric acid solutions that had been filtered off showed about 0.06 per cent of phosphoric acid.

Another set of samples was run according to method B, only a larger excess of citric acid was used in the digestion. The citric acid solutions upon being analyzed showed 1.40, 1.66, and 1.48 per cent of phosphoric acid. The residues showed 6.13, 5.71, and 5.40 per cent of citrate-soluble or reverted phosphoric acid, making the available phosphoric acid in this case 7.53, 7.37, and 6.93 per cent. It was noticed when the residue from the citric acid solution was being washed with hot water, that after a few washings the water, as it left the residue, was of a pinkish color.

The sample of slag on which the above tests were made showed 47.83 and 47.72 per cent or an average of 47.77 per cent of calcium oxid of which 7.38, 7.40, and 7.43 per cent were actually found present as free calcium oxid.

No attempt was made to determine the available phosphoric acid in the above slag by the method in use at the California experiment station or by the Wagner method. It appears to the writer that of the two methods, Method B offers the most encouragement for development. Although not giving satisfactory concordant results, as above outlined, it would seem that if a given quantity of solution of citric acid of known strength were used in each case, satisfactory comparative results would follow. It is earnestly hoped that the association will investigate the matter of the determination of the available phosphoric acid in basic slag and adopt, at least provisionally, some method for the analysis of this product, the use of which is becoming general in certain sections of our country.

REPORT OF COMMITTEE B ON RECOMMENDATIONS OF REFEREES.

B. B. Ross, *Chairman*.

(Dairy products, foods and feeding stuffs, sugar, tannin, and medicinal plants and drugs.)

(1) SUGAR.

It is recommended—

(1) That in the determination of reducing sugars the use of lead subacetate as a clarifying agent be discontinued and that only those agents, such as normal lead acetate, be employed which do not precipitate reducing sugars.

Adopted.

(2) That the use of potassium oxalate for precipitating the excess of lead from sugar solutions before polarizing or determining reducing sugars be adopted provisionally.

Adopted.

(3) That the zinc oxid method for the determination of ash in molasses be dropped from the official methods. (Bul. 107, p. 68, Method II.)

Adopted.

(4) That the method and tables of Munson and Walker for the unification of reducing sugar methods be adopted provisionally by the association. (J. Amer. Chem. Soc., 1906, 28: 663; 1907, 29: 541.)

Adopted.

(5) That Low's volumetric method for the estimation of reduced copper (Bul. 99, p. 19) be made a provisional method of the association.

Adopted.

(6) That the question of the influence of normal lead acetate, of wet and dry lead subacetate, and of hydrosulphite upon the polarization of sugars be further investigated.

Adopted.

(7) That the analysis of commercial dextrans be further investigated by next year's referee on special analytical methods.

Adopted.

(2) FOODS AND FEEDING STUFFS.

It is recommended—

(1) That the method for methyl pentosans be further studied next year, especial attention being given to obtaining as much cooperative work as possible.

Adopted.

(2) That the table for calculating methyl-furfural-phloroglucid to fukose, fukosans, rhamnose, rhamnosans, and methyl-pentosans (average of fukosan and rhamnosan), given in an article by Mayer and Tollens (J. Landw., 1907, 55 (3): 269), be used in calculating the results of analysis; the average figure for methyl pentosans being taken when it is not known whether the mother product is fukosan or rhamnosan.

Adopted provisionally.

(3) That the last sentence under "Crude Fat or Ether Extract," "(b) (1) Direct Method," Bulletin 107, Methods of Analysis, page 39, which now reads as follows: "Dry the extract at the temperature of boiling water until it ceases to lose weight," be changed to read: "Dry the extract at the temperature of boiling water for one-half hour, remove from the oven to a desiccator, cool and weigh; continue this alternate drying and weighing at half-hour intervals until a minimum weight of fat is obtained. For most feeds a period of from one to one and one-half hours is required to obtain a minimum weight."

Adopted. [This is an elaboration of an official method and was adopted as a recommendation of the chairman of the committee on revision of methods, ordinarily two years being necessary for such action.]

(3) DAIRY PRODUCTS.

It is recommended—

(1) That the study of methods of analysis of condensed milk be continued and that special attention be given to the following methods for determining the fat and sugar content of condensed milk:

(a) Double extraction method for the determination of fat as described in the proceedings of the association for 1906.^a

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 105, p. 105.

(b) Roese-Gottlieb method for the determination of fat as described in the proceedings of the association for 1906.^a

(c) Walker method for the determination of sugar. (J. Amer. Chem. Soc., 1907, 29: 541.)

(d) Allihn method for the gravimetric determination of sugar.

(e) Polariscopic method in comparison with the gravimetric method for the determination of sugar.

Adopted.

(4) TANNIN.

It is recommended—

(1) That work be continued with a view to finding a cheap strong filter paper as a substitute for S and S 590.

(2) That the method of filtering soluble solids suggested by Reed be given further trial. (J. Amer. Leather Chemists Assn., 2: 420.)

(3) That the referee take up the study of methods for the analysis of leather, as such methods are greatly needed.

Recommendations adopted.

(5) MEDICINAL PLANTS AND DRUGS.

It is recommended—

(1) That the present provisional method for assaying opium be made official. (Bul. 107, p. 201.)

Referred to referee for recommendation as to final action in 1908.

(2) That the methods included in the referee's report be made provisional. (See p. 81.)

Adopted.

(3) That the work be continued and that the associate referees be appointed on the following subjects:

(a) Analytical methods for crude products.

(b) Methods for detecting and estimating alkaloidal matter in mixtures.

(c) Macroscopical and microscopical methods.

(d) Microchemical methods.

(e) Methods for headache mixtures.

(f) Methods for determining alcohol in the presence of other volatile bodies.

(g) Methods for testing volatile oils.

After some discussion this motion was put to a vote and was lost, it being deemed inadvisable to create associate referees for such divisions of the subject at this time, and the referee was instructed to conduct the investigations specified informally, assigning the several lines of work to such chemists as volunteered for cooperative work.

A motion was made to continue the committee for the testing of chemical reagents another year, and this motion was carried.

Mr. Kebler called attention to the fact that it was often desirable to use the text of the United States Pharmacopœia in connection with the association work on drugs, and this could not be done without the consent of the trustees of the Pharmacopœia. It was moved, therefore, that the secretary of the association be authorized to correspond with the proper authorities and secure this privilege. The motion was carried.

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 105, p. 105.

REPORT ON POTASH.

By A. L. KNISELY, *Referee*.

At the last meeting of the association it was recommended that the study of the volumetric method for use in both soil and fertilizer analyses be continued. It was also recommended that the referee direct his attention to a study of what really constitutes available potash in soils, fertilizers, and ground mineral products, to the end that available potash might be defined.

The referee, assisted by Mr. C. E. Bradley, spent all of the time possible upon a study of the volumetric method, leaving the subject of available potash for consideration during the coming year. Considerable time was spent in preliminary experimental work upon details of the method before a satisfactory scheme was evolved as an outline for cooperative work.

In applying the method of Donk for estimating potash volumetrically, approximately correct results can be readily obtained. At times, however, discrepancies were noted in comparing volumetric values with those obtained by gravimetric methods. One of the vital points in the process is the use of a wash liquid which will remove all excess acid from the precipitated potassium phosphomolybdate and at the same time dissolve only imprecipitable quantities of this yellow precipitate. Accordingly a test was made to determine the solubility of the potash precipitate in both nitric acid and sodium nitrate under different conditions in respect to strength, time of contact, etc. For this purpose a quantity of pure potassium phosphomolybdate was prepared by adding phosphomolybdic acid to excess of potassium sulphate in solution and washing the precipitate with a weak solution of sodium nitrate. To determine the action of nitric acid upon this precipitate, 0.1 gram of potassium phosphomolybdate was placed in contact with 50 cc of the nitric acid of varying strength, well stirred and filtered. The filtrate was evaporated to dryness on a water bath and the residue titrated with potassium hydroxid.

TABLE I.—*Determination of the solubility of the potash precipitate in nitric acid.*

No.	Conditions.	Strength of nitric acid.	Amount of potassium hydroxid required to neutralize.
			cc.
1	Heated to 50° C. and cooled 1 hour.....	5.5:1,000	1.00
2	One hour in cold.....	5.5:1,000	.10
3	Five minutes in cold with constant stirring.....	40 :1,000	.12
4	Ten minutes in cold with stirring.....	80 :1,000	.20

Heating to 50° C. is thus seen to exert marked solvent action, even with very dilute solutions of nitric acid. The cold acid of much greater strength exerts less solvent action, when time of contact is limited.

The action of the nitric acid wash under different conditions was also tested on an official acid (sp. gr. 1.115) extract of soil. Ten cubic centimeters of solution, corresponding to 1 gram soil, were evaporated with phosphomolybdic acid and the residue subjected to the following methods of washing:

TABLE II.—*Determination of the solubility of the precipitate of an official acid extract of soil in nitric acid.*

Sample.	Conditions.	Strength of nitric acid.	Potassium monoxid.
			<i>Per cent.</i>
1.....	Stirred in cold 5 minutes.....	5.5:1,000	0.41
2.....	Heated to 50° C. and cooled 30 minutes.....	5.5:1,000	.35
3.....	Heated 50° C. for 30 minutes and filtered after 5 hours.....	5.5:1,000	.23
		5.5:1,000	.24

The solubility of potassium phosphomolybdate in sodium nitrate solutions under different conditions was also investigated. A weighed quantity of the potassium phosphomolybdate was transferred to a gooch and 250 cc of sodium nitrate wash were run through as in analysis. The precipitate remaining in the gooch was then titrated with potassium hydroxid solution, 1 cc of the alkali used being equivalent to 0.0298 gram potassium phosphomolybdate.

TABLE III.—*Solubility of potassium phosphomolybdate in sodium nitrate.*

Sample.	Strength of sodium nitrate.	Phosphomolybdate.	
		Used.	Found.
		<i>Grams.</i>	<i>Grams.</i>
1.....	5:1,000.....	0.1461	0.1474
2.....	20:1,000.....	.0915	.0920

A negative result for solubility was thus obtained, probably due to a small error in the factor previously obtained. It is evident that a sodium nitrate wash of 2 per cent strength exerts very little solvent action on the precipitate of potassium phosphomolybdate under the conditions of the experiment. On long contact, however, a marked solvent action is evidenced by the yellow color which a solution develops when in contact with the precipitate. A test of a saturated solution gave results close to those noted by Fraps (1905).

It was also noted that pure water decomposes the precipitate, as stated by Donk, with the separation of a whitish acid substance, supposedly molybdic acid. In our hands a solution of sodium nitrate of the strength adopted (5:1,000) gave also a slight turbidity when left in contact with the precipitate for a short time. A solution of sodium nitrate (10:1,000) remains clear, however, under these conditions.

The preparation of the phosphomolybdic solution was also investigated, as it was noted that some solutions left no trace of insoluble residue when evaporated and taken up with nitric acid wash, while others left molybdic acid residues on evaporating, which required from 0.2 cc to 3 cc of standard potassium hydroxid solution to neutralize and which had to be allowed for as a "blank." Crystals of phosphomolybdic acid as furnished by Kahlbaum left no residue when the water solution was evaporated on a water bath and the dry residue again taken up with water. The nitric acid solution of the crystals, as recommended by Donk, when fresh left no residue under these conditions, but when allowed to stand some weeks left a residue from the nitric wash, which gradually increased in amount as the solution became older. The addition of a small amount of calcium and magnesium nitrate in the presence of a large excess of nitric acid seemed to prevent this, probably due to the formation of

soluble molybdates of calcium and magnesium with the separated molybdic acid. The presence of hydrochloric acid also prevented the formation of an insoluble residue, the hydrochloric acid combining with the molybdenum trioxid and forming a compound soluble in water or dilute nitric acid.

Phosphomolybdic acid solutions were prepared as follows:

- I. { 100 grams phosphomolybdic acid.
250 cc nitric acid (1.40 sp. gr.).
750 cc water.
- II. Duplicate of I (kept in dark).
- III. { 100 grams phosphomolybdic acid.
250 cc nitric acid (1.40 sp. gr.).
750 cc water.
10 grams calcium nitrate.
10 grams magnesium nitrate.
- IV. { 100 grams phosphomolybdic acid.
200 cc nitric acid (1.40 sp. gr.).
100 cc hydrochloric acid (1.20 sp. gr.).
700 cc water.
- V. { 100 grams phosphomolybdic acid.
40 cc nitric acid (1.40 sp. gr.).
960 cc water.
10 grams calcium nitrate.
10 grams magnesium nitrate.

These solutions were kept on the laboratory desk in rubber-stoppered Erlenmeyer flasks, with the exception of No. II, which was kept in the dark. From time to time 10 cc of each of these solutions were evaporated in beakers on the water bath to complete dryness—that is, until the odor of acid had completely disappeared. An examination of the dry residue taken up in the cold with nitric acid wash (50:1,000) resulted as follows:

TABLE IV.—*Solubility of residue from 10 cc phosphomolybdic acid solution in nitric acid (50:1,000).*

Solution.	July 24.	August 6.	August 20.	September 26.	October 3.	October 4. [Standard potash neutral- izing residue.]
I	Clear....	Trace precipitate	Trace precipitate	Small precipitate	Small precipitate.	cc. 0.10
II	Clear....	Trace precipitate	Small precipitate	Small precipitate	Small precipitate.	.10
III	Clear....	Clear.....	Clear.....	Clear.....	Clear.....	.00
IV	Clear....	Clear.....	Clear.....	Clear.....	Clear.....	.00
V	Clear....	Trace precipitate	Considerable white precipitate.	Heavy white precipitate.	Heavy white precipitate.	.70

These results show that much depends upon the age of the phosphomolybdic acid solution and upon the method of preparation. Fresh solutions are always to be preferred.

The accuracy of the method proposed for this year was checked by testing chemically pure potassium sulphate, with the result that in a sample containing theoretically 0.1284 gram of potassium monoxid 0.1279 gram was found, and 0.0398 gram was found as compared with 0.0399 gram present according to theory.

The evaporated residue from chemically pure potassium sulphate and phosphomolybdic acid is a fine amorphous powder which gave difficulty in filtering. The addition of a small amount of calcium or magnesium nitrate assisted in the formation of a crystalline residue which facilitated filtering greatly.

The presence of a soluble silica in soil extract did not interfere with the process, except that when the amount was large, filtration became troublesome from the clogging of the filter.

The use of the centrifuge for freeing the yellow precipitate of potassium phosphomolybdate from acid was tried, with good results. The use of a potassium hydroxide solution (1 cc = 1 mg K_2O) is probably preferable, but to avoid the labor of preparing two solutions the potassium hydroxide used in volumetric phosphoric acid work was utilized. As a result of all this preliminary work directions were formulated which gave good results in the station laboratory. The samples and directions were sent out as follows:

ASSOCIATION POTASH WORK, 1907.

Sample No. 1.—A sample of soil from the wheat-growing belt of eastern Oregon. This soil contains approximately from 0.25 to 0.5 per cent of acid-soluble potash.

Sample No. 2.—A mixed fertilizer containing considerable quantities of nitrogen, phosphoric acid, and potash (approximately 9 to 10 per cent of potassium monoxide).

WORK ON SOIL.

Mix thoroughly before sampling.

(1) Determine potash (K_2O) in soil according to the official method using hydrochloric acid, 1.115 sp. gr. (U. S. Dept. Agr., Bureau of Chemistry, Bul. 46 Revised or Bul. 107).

(2) Proposed volumetric method for potash in soils.

Reagents.

Nitric acid.—50.0 cc nitric acid 1.40 sp. gr. in 1,000 cc water.

Sodium nitrate wash.—10.0 grams sodium nitrate per 1,000 cc water.

Phosphomolybdic acid solution.—100 grams phosphomolybdic acid (Kahlbaum's preferred) in 750 cc water and 250 cc nitric acid (1.40 sp. gr.). This solution must be freshly prepared—not over three or four days old before using. If properly prepared, the evaporated residue from a portion of this solution is never white and readily redissolves in the dilute nitric acid solution in the cold.

Standard solutions.—Standard caustic potash and nitric acid prepared for volumetric phosphoric acid; 1 cc potassium hydroxide is equal to 1.625 mg K_2O .

Determination.

Place a fresh aliquot, representing 1 gram of soil of solution A under soil analysis (Bul. 107 Revised, p. 14), in a tall 200 cc beaker, add 20 cc phosphomolybdic solution, and slowly evaporate to complete dryness on a steam bath. If preferred, use solution without removal of silica, evaporating the acid soil solution directly with phosphomolybdic acid. (It requires approximately 22 mg of phosphomolybdic acid in order to have an excess for each milligram of potassium monoxide present.)

Add 30 cc of nitric acid wash to the dried residue, stir thoroughly in the cold with a grinding motion, using a policeman, allow to settle a moment, and decant supernatant liquid at once through a gooch crucible packed with moist filter-paper pulp approximately one-sixteenth inch in thickness. Wash twice by decantation with sodium nitrate wash, transfer precipitate to gooch, and wash with sodium nitrate wash until acid-free. Transfer gooch to casserole, run in excess of standard alkali solution, and add phenolphthalein. Heat to boiling and titrate excess of alkali with standard acid.

Note.—Some samples of asbestos used seemed to hold or "fix" some of the excess acid, making it very hard to wash the gooch filter acid-free. Hence it is suggested to use a paper-pulp filter.

It is suggested that the evaporated residue in the beaker be transferred to a centrifuge tube instead of a gooch, using nitric acid wash, whirl until well settled, decant, and wash with sodium nitrate wash, using the centrifuge, until acid-free. Dissolve in standard alkali and titrate as above.

WORK ON MIXED FERTILIZERS.

Mix thoroughly before sampling.

(1) In sample No. 2 determine water-soluble potash (K_2O) by the official method (U. S. Dept. Agr., Bureau of Chemistry Bul. 46 Revised, p. 21, or Bul. 107, p. 11).

(2) Use proposed volumetric method for potash in fertilizers. Transfer 10 cc of solution No. 2 (Bul. 46 Revised, pp. 21-22, or Bul. 107, pp. 11-12) to a platinum dish, and add 0.25 cc of sulphuric acid (1 to 1). Evaporate to dryness and ignite to whiteness. Dissolve residue in hot water plus a few drops of hydrochloric acid and transfer to a tall 200 cc beaker; add 30 cc of phosphomolybdic acid solution and proceed as under proposed volumetric method for potash (K_2O) in soils. (If excess of phosphomolybdic acid has been used, the dried residue has a reddish hue. If excess has not been added, the residue is bright yellow. The residue should not appear white.)

In each case run blanks to ascertain corrections to be made for impurities. It is also advisable to treat the potassium-platinic-chlorid residue in the gooch crucible in order to ascertain if it is all soluble in water. Then reweigh the crucible after thoroughly drying.

Note.—The caustic potash solution must be free from carbonate. (See Sutton, Volumetric Analysis, 9th edition, revised, page 48, for the barium hydrate method, also page 301 for the alcoholic method for making potassium hydroxid free from carbonates.)

A. L. KNISELY, *Referee*.

B. B. ROSS, *Associate Referee*.

Ten sets of samples were sent out and three reports have been received.

TABLE V.—Comparison of official and proposed volumetric method for the determination of potash in soil and in mixed fertilizers.

Analyst.	Soil No. 1.		Fertilizer No. 2.		
	Official.	Volumetric.	Official water-soluble.	Volumetric.	Official—burning off with $(NH_4)_2SO_4$ instead of H_2SO_4 .
M. G. Donk, Bureau of Chemistry, Washington, D. C.	<i>Per cent.</i> 0.35 .37 .39 .394243	<i>Per cent.</i> 0.44 .38 .40 .464243	<i>Per cent.</i> 10.59 10.66 10.62	<i>Per cent.</i>	<i>Per cent.</i> 10.55 10.53 10.66
A. L. Knisely and C. E. Bradley, Oregon station	.42 .42 .42	.39 .41 .38	10.65 10.71	10.82 10.65	
S. E. Asbury, Texas station	<i>a</i> .50 <i>a</i> .55	.40 .52 .45 .35	<i>a</i> 11.11 <i>b</i> 11.14 <i>a</i> 11.19 <i>b</i> 11.09	11.62 11.89 11.92	
E. Carlyle, Texas station					

a Direct weight.

b Washed out.

c By volumetric method formerly used.

d No correction for reagent used.

e Washed by centrifuge.

COMMENTS BY ANALYSTS.

M. G. Donk: Could obtain no satisfactory results on sample No. 2 with the volumetric method, presumably due to difficulty in washing yellow precipitate.

G. S. Fraps: Sample of phosphomolybdic acid from Merck left considerable white residue, probably molybdic acid, when diluted in nitric acid. This probably accounts for volumetric results being higher than the official method.

C. E. Bradley: Best results were always obtained when using small amounts of potash solution. An aliquot part of a solution of fertilizer containing not more than 0.02 gram K_2O gave the best results. If, after evaporating to dryness and taking up in dilute nitric acid, the insoluble residue is not bright yellow, then the conditions are not normal. The washed precipitate should be bright yellow.

COMMENTS BY REFEREE.

It was recommended this year to use a stronger nitric acid wash solution than was formerly used. This was on account of the difficulty experienced in washing the yellow precipitate free from impurities. It has since been ascertained that this difficulty was due to poorly prepared or old phosphomolybdic acid solution. The phosphomolybdic acid solution, when prepared from good, fresh chemicals, should give no blank, and when used in volumetric potash work dissolves and washes out very readily from the evaporated residue. It has recently been found that the nitric acid wash, instead of being stronger than was formerly used, should, if anything, be weaker. In some cases when determining potash in material containing considerable calcium and magnesium and moderate quantities of iron, such as some soil solutions, it is not necessary to use a nitric acid wash solution, but only to dissolve the material at once in the sodium nitrate wash solution.

Official soil solution evaporated to dryness with phosphomolybdic acid and taken up in nitric acid wash gave 0.45 per cent K_2O ; when taken up directly in sodium nitrate wash the same result was obtained.

I think the merit of the volumetric method for potash depends entirely upon the phosphomolybdic acid solution and the care with which the evaporation is conducted. The difficulties thus far experienced with blanks and in washing the evaporated residues have been largely due to the presence of white insoluble molybdic acid, which at times separates during the process of evaporation and is difficult to remove from the yellow residue of potassium phosphomolybdate.

RECOMMENDATION.

The referee recommends a continuation of the study of the volumetric method for use in both soil and fertilizer analysis; also that the recommendation of Mr. Cushman concerning a study of what really constitutes available potash in soils, fertilizers, and ground-mineral products be continued, so that if possible we may have a definition of available potash.

REPORT ON INSECTICIDES.

By R. J. DAVIDSON, *Referee*.

The suggestions made in the recommendations of last year were followed as closely as possible, and after consultation with the associate referee the following work was outlined:

(1) Work on London purple to test the Davidson modification of Haywood's method in comparison with the method as originally outlined.

(2) Tests to determine which of the two methods of analysis of soda lye give the more correct results.

(3) Testing the formaldehyde method as modified by Haywood and Smith, in comparison with the method as it originally stood.

(4) Testing the Avery method of determining sulphur in sulphur dips.

(5) Testing the methods of examining lead arsenate as published in last year's proceedings.

Requests for cooperation were sent to thirty chemists. Of these, but two signified their willingness to undertake the work. One report contained results obtained by two analysts in the same laboratory. The associate referee was prevented from doing any work on account of a serious injury he received as the result of an accident in his laboratory. The report therefore contains the results of five analysts.

LONDON PURPLE.

The work on London purple consisted in testing the modified Haywood method (for the removal of part of the coloring matter) in comparison with the method as originally outlined. The modification was proposed in 1903 and was given a partial trial in 1904, but no definite results were obtained. A statement was made that it greatly facilitated end point readings. The benefits of this modification are twofold: Time is saved and, by removing a part of the coloring matter, the end point reading in the titration is made less difficult. If, therefore, the modification does not interfere with its accuracy, it will be of great advantage.

In presenting the work done it has been deemed advisable to give the results for arsenious and arsenic oxids in separate tables, as the work on these two determinations has not been equally satisfactory. The subject will therefore be discussed under two heads.

TOTAL ARSENIOS OXID.

Method I is a provisional method, and may be found in Circular No. 10, Revised, Bureau of Chemistry.

Method II is the proposed modified method, and differs from Method I in that the process of filtering and washing to a definite volume after dissolving the material is eliminated, and in addition a part of the coloring matter is precipitated. The method is as follows:

Place 2 grams of London purple in a beaker and dissolve in about 80 cc of water and 20 cc of concentrated hydrochloric acid at a temperature of 60° to 70° C.; cool, and add sodium carbonate in slight excess, transfer to a 250-cc flask, bring to mark, shake, and filter through a dry filter. Acidify 50 cc of the filtrate with hydrochloric acid and make alkaline with sodium bicarbonate. Titrate the amount of arsenious oxid present with standard iodine solution.

TABLE I.—Total arsenious oxid.

Analyst.	Method I.	Method II
	<i>Per cent.</i>	<i>Per cent.</i>
R. W. Thatcher, Pullman, Wash.	16.78	16.43
	16.94	16.50
	16.86	16.47
H. R. Watkins, Pullman, Wash.	16.83	16.41
	16.83	16.67
	16.83	16.54
J. K. Haywood and C. C. McDonnell, Bureau of Chemistry.	16.54	16.33
	16.54	16.25
	16.72	16.33
R. R. Henley, Blacksburg, Va.	16.72	16.22
	16.70	16.31
R. J. Davidson, Blacksburg, Va.	16.75	16.54
	16.70	16.35
	16.75	16.54
Average.	16.75	16.42

Discussion.

The results upon total arsenious oxid agree very well. There is a difference of only 0.33 per cent between the averages by the two methods, showing that

in case of the total arsenious oxid, the shorter method gives practically the same results.

TOTAL ARSENIC OXID.

Methods I and II are the same as were used in 1904 and 1905, and may be found in Circular No. 10, Revised, of the Bureau of Chemistry. Method II is a modification of Method I, the difference being that before proceeding with the reduction of the solution a part of the coloring matter is removed by precipitation with sodium carbonate. Method III is a still further modification of Method I, as will be seen by reference to Method II under total arsenious oxid.

Method III reads as follows:

Acidify 50 cc of the solution prepared under determination of total arsenious oxid, Method II, with concentrated hydrochloric acid, heat to 80° C., add 50 cc more concentrated hydrochloric acid and 3 grams of potassium iodid, and proceed as described in Method I for total arsenic oxid.

The only difference between Methods II and III is the elimination of the filtering and washing to definite volume of the hydrochloric acid solution, which saves considerable time

TABLE II.—Total arsenic oxid.

Analyst.	Method I.	Method II.	Method III.
	Per cent.	Per cent.	Per cent.
R. W. Thatcher, Pullman, Wash.....	12.43	11.04	9.73
	12.12	11.15	10.09
	12.27	11.10	9.86
H. R. Watkins, Pullman, Wash.....	12.91	9.93	9.55
	13.06	10.08	9.45
	12.98	10.00	9.50
J. K. Haywood and C. C. McDonnell, Bureau of Chemistry.....	13.15	13.05	12.98
	13.25	13.25	12.98
	12.95	12.95	12.90
R. R. Henley, Blacksburg, Va.....	12.67	10.14	12.94
	12.98	10.34	9.86
	12.52		10.43
R. J. Davidson, Blacksburg, Va.....	12.66		
	12.95	10.35	10.75
	12.51		9.96

Discussion.

The results upon total arsenic oxid are very unsatisfactory. There is a fairly good agreement of the results of four analysts under each method, but the results of the different methods do not agree, there being a difference of about 2 per cent between Method I and Methods II and III. One set of results shows an excellent agreement by all three methods. All of the results agree very well by Method I, but by Methods II and III one set of results is about 2 per cent higher than the other four.

It is very difficult, if not impossible, to explain why four sets of results should agree so well among themselves by Methods II and III, yet differ so widely from the other set. If there had been agreement between all the analysts, then the difference between Method I and Methods II and III might be attributed to one of two things: an error in Method I due to the difficulty of reading accurately the end point in the titration when so much coloring matter is present, or to the loss of arsenic occurring in removing the coloring matter. It was suggested that the difference was partly due to not carrying out properly the details of Method III, as it was thought that the directions for Method III were not explicit enough as to heating the solution before adding

hydrochloric acid and using only 25 cc instead of 50 cc. This can not be the explanation, for in my own work the solution was heated to 80° C. and 50 cc concentrated hydrochloric acid tried as well as 25 cc, giving practically the same results. It is regretted that no comments are made by the analysts on this point, and also that the referee did not have time to investigate it further. Method II was used in 1903 and 1904, but no mention was made of any difficulty occurring; in fact, the referee stated in his discussion that "the modification suggested for the removal of a part of the coloring matter works very well, greatly facilitating the end point reading." In applying this method to the samples of 1903, I did not notice such a difference in results. This part of the work on London purple must be repeated next year, and it is to be hoped that the cause of the discrepancy will be discovered.

SODA LYE.

The methods used for the examination of soda lye were the same as have been used for several years, and may be found in Circular No. 10, Revised, Bureau of Chemistry.

In Method I the total alkalinity is obtained by titrating an aliquot of the dilute solution with half-normal acid, using methyl orange as an indicator. Another aliquot is taken, and the carbon dioxide of the carbonate is precipitated by a dilute solution of barium chloride and then made to a definite volume. The precipitate is allowed to settle, and an aliquot of this is taken and titrated with half-normal acid, using phenolphthalein as an indicator. In Method II an aliquot is titrated with acid potassium sulphate, using phenolphthalein and methyl orange as indicators.

TABLE III.—*Soda lye.*

Analyst.	Method I.		Method II.	
	Sodium hydroxid.	Sodium carbonate.	Sodium hydroxid.	Sodium carbonate.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
R. W. Thatcher, Pullman, Wash.....	88.91	5.29	91.53	5.44
H. R. Watkins, Pullman, Wash.....	89.12	5.00	90.80	5.13
J. K. Haywood and C. C. McDonnell, Washington, D. C. {	91.82	3.30	91.25 91.15	4.18 4.18
	91.71	2.74		
	91.43	2.75		
R. R. Henley, Blacksburg, Va.....	91.46	3.56	91.59	4.67
	92.09	3.93		
R. J. Davidson, Blacksburg, Va.....	91.73	3.56	91.04	4.70
	92.04	3.40		

DISCUSSION.

The results are not so good as they have been in previous years. Two sets of results are slightly lower in sodium hydroxid and higher in sodium carbonate than the others. The results for sodium hydroxid by the two methods agree very well. The sodium carbonate by Method II is higher. This higher result may be due to the difficulty in reading the end point with methyl orange. In Method II the titration is tedious, and for this reason, together with the difficulty in reading the end point in the titration, the referee thinks it should be dropped from the methods. In Method I, when titrating with phenolphthalein to determine the sodium hydroxid after precipitating with barium chloride, it is not necessary to filter off the precipitate, as has been pointed out before, since the precipitate does not interfere with the titration. The results obtained when the precipitate is permitted to remain in the solution are the same as when it has been removed. Better results would probably be obtained in this work if a fifth normal solution of hydrochloric acid were used instead of half normal. A

difference of 0.1 cc of half-normal solution makes a difference of 0.6 per cent in the result.

FORMALDEHYDE.

The methods used for the analysis of formaldehyde were the modified hydrogen peroxid method proposed by Mr. Haywood and the cyanid method.

TABLE IV.—*Formaldehyde.*

Analyst.	Hydrogen peroxid method.	Potassium cyanid method.
	<i>Per cent.</i>	<i>Per cent.</i>
R. W. Thatcher, Pullman, Wash.....	36.24	35.24
	36.42	35.24
	36.33	35.24
	36.24	34.50
H. R. Watkins, Pullman, Wash.....	36.15	34.79
	36.20	34.65
	36.00	
J. K. Haywood and C. C. McDonnell, Bureau of Chemistry.....	36.18	
	36.15	
	36.30	35.22
R. R. Henley, Blacksburg, Va.....	36.30	35.22
R. J. Davidson, Blacksburg, Va.....	36.30	35.20
	36.30	35.21

COMMENTS OF ANALYSTS.

J. K. Haywood.—The results on formaldehyde are no doubt lower than they would have been had the determination been made when the sample was received, as some insoluble polymerized formaldehyde settled on the bottom of the bottle during the time intervening between its receipt and its analysis. The acidity of the hydrogen peroxid used was determined and correction made therefor.

DISCUSSION.

The results as a whole are exceedingly good. Method II can only be used with a solution containing a small quantity of formaldehyde; therefore it seems advisable to state the strength of the solution more definitely—for example, about 1 per cent or 2 per cent solution, instead of specifying “a small quantity.” This point is referred to the next referee for investigation, and it is recommended that the hydrogen peroxid method be made official.

SULPHUR DIPS.

The method for sulphur dips was the same as that tested by the referee in 1905, and is given in Circular No. 10, Revised, Bureau of Chemistry.

TABLE V.—*Sulphur dips.*

Analyst.	Month of analysis.	Weight of sulphur.	Sulphur by weight.
		<i>Gram per cc.</i>	<i>Per cent.</i>
R. W. Thatcher, Pullman, Wash.....	June.....	0.0484	4.61
		.0484	4.61
		.0484	4.61
		.0456	4.35
H. R. Watkins, Pullman, Wash.....	June.....	.0455	4.34
		.0456	4.36
		.0465	
J. K. Haywood and C. C. McDonnell, Washington, D. C.....	June.....	.0476	
		.0466	
		.0416	
R. R. Henley, Blacksburg, Va.....	June.....	.0427	
R. J. Davidson, Blacksburg, Va.....	June.....	.0431	
		.0435	

DISCUSSION.

The results are all good, the difference between the lowest and the highest being about 0.5 per cent. The method is easy of manipulation and very satisfactory. I would recommend that it be adopted as an official method.

LEAD ARSENATE.

The methods used for lead arsenate were those proposed by Mr. Haywood at the last meeting of the association, and are found in the Proceedings of 1906.^a

The material used for analysis was purchased from the same person from whom the other samples used in this work were obtained, and when received was in the form of a thick paste. It was dried and ground before being sent out.

TABLE VI.—*Lead arsenate.*

Analyst.	Moisture.	Total arsenic oxid.	Total lead oxid.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
R. W. Thatcher, Pullman, Wash.....	1.37	25.57	65.10
	1.40	25.72	64.83
	1.38	25.64	64.95
	1.50		64.49
H. R. Watkins, Pullman, Wash.....	1.50	25.31	64.34
	1.50		64.95
			66.70
J. K. Haywood and C. C. McDonnell, Washington, D. C.....	1.18	24.74	66.73
		24.57	66.01
		24.40	66.88
		24.57	65.40
R. R. Henley, Blacksburg, Va.....	1.00	24.41	65.40
R. J. Davidson, Blacksburg, Va.....	1.00	24.60	65.40
		24.44	65.72
		24.66	65.44

DISCUSSION.

The results are not as good as they should be. On total lead oxid there is a difference of over 2 per cent between the lowest and highest, and on the total arsenic oxid the difference is over 1 per cent. I would recommend that these methods be further tried by the next referee.

RECOMMENDATIONS.

It is recommended—

(1) That the Haywood-Davidson provisional method for determining total arsenic oxid in London purple be given a further trial, and that this be compared with the original provisional method.

(2) That a further study of the amount of hydrochloric acid used in the reduction of London purple be made in order to determine if 25 cc can be used instead of 50 cc.

(3) That the provisional precipitation method for the analyses of soda lye be referred to the referee with a view to its adoption as official.

(4) That the provisional potassium acid sulphate method for the analysis of soda lye be dropped from the methods of analysis.

(5) That the provisional hydrogen peroxid method for the determination of formaldehyde be referred to the referee with a view to its adoption as official.

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 105, page 165.

(6) That the cyanid method for the determination of formaldehyde be further studied in order to determine more definitely what strength of formaldehyde may be estimated by this method.

(7) That the provisional hydrogen peroxid method for the determination of sulphur in sulphur dips be referred to the referee with a view to its adoption as official.

(8) That the methods proposed by Mr. Haywood for the analyses of lead arsenate be made provisional and further studied with a view to their adoption by the association.

REPORT OF COMMITTEE A ON RECOMMENDATIONS OF REFEREES.

By R. J. DAVIDSON, *Chairman.*

(Phosphoric acid, potash, nitrogen, soils, ash, and insecticides.)

(1) NITROGEN.

It is recommended—

(1) That "20 cc of sulphuric acid," line 3, under (3) Determination, page 6, Bulletin 107, on Methods of Analysis, be changed to read "from 20 to 30 cc of sulphuric acid."

Adopted.

(2) That in line 4, same reference, the following phrase be inserted after the word "acid:" "from 0.1 to 0.3 gram of crystallized copper sulphate may also be used in addition to the mercury or in lieu of it."

Referred to the referee for 1908 with a recommendation for adoption.

(3) That following the sixth line from the bottom of page 6, same reference, after the words "standard acid," this sentence be added: "During at least the first ten minutes of distillation the lower end of the condenser tube should dip beneath the surface of the standard acid."

The motion to adopt this recommendation was lost.

(4) That on page 7 of Bulletin 107, under "(b) Gunning method, (3) Determination," line 4, after the words "sulphuric acid" the following phrase be inserted: "from 0.1 to 0.3 gram of crystallized copper sulphate may also be added."

Referred to the referee for final action as to adoption in 1908.

(5) That on page 7 of Bulletin 107, after insert recommended under (4), add the following:

Approximately 0.7 gram of mercuric oxid or its equivalent in metallic mercury may also be added, before the addition of the potassium sulphate, but if mercury be used potassium sulphid must be employed, as in the Kjeldahl method, in the distillation.

This recommendation was referred to the committee on the revision of methods to be inserted as a special method instead of being introduced as the referee recommended.

(2) POTASH.

It is recommended—

(1) That since the volumetric method for potash, especially in soils, saves much time, the study of this method for use in soil and fertilizer analysis be continued; and further that the recommendation made by Mr. Cushman in 1906, to the effect that the referee "turn his attention to a definition of available potash," be made the basis for work the coming year.

Adopted.

(3) PHOSPHORIC ACID.

It is recommended—

(1) That Thomas or basic slag be prepared for analysis as are other fertilizers or fertilizing materials under the methods of this association.

Adopted provisionally pending further work.

(2) That the solution for analysis be made according to (a₇), page 2, Bulletin 107, under "(2) Total phosphoric acid," or in strong hydrochloric acid alone. In the latter case, after the portion for analysis is measured out, add nitric acid and heat for a few minutes. Then determine total phosphoric acid according to the official methods.

Adopted provisionally pending further work.

(3) That the fineness of the material be determined according to the plan followed with bone meal and the commercial value estimated on the basis of total phosphoric acid and fineness of product.

Adopted provisionally pending further work.

(4) That the referee further study the subject of basic slag with a view to devising some method for the determination of the available phosphoric acid which it contains.

This recommendation was made by Committee A and was adopted.

(4) INORGANIC PLANT CONSTITUENTS.

It is recommended—

(1) That the peroxid method remain provisional [not adopted as official as recommended by the referee].

Adopted.

(2) That the combustion method be adopted as provisional.

Adopted.

(3) That the method for ash, as given in Bulletin 46, page 77, be readopted as official.

Adopted.

(4) That the development of methods for determining iron and aluminum in ash be referred to the referee for 1908.

Adopted.

(5) SOILS.

It is recommended—

(1) That the modified J. L. Smith method for total potassium be further tested. (Circular 32, p. 4.)

Adopted.

(2) That the sodium peroxid fusion method for total phosphorus be adopted as a provisional method of this association and be further tested. (Bul. 99, p. 111; Bul. 105, p. 145.)

Adopted.

(3) That the determination of volatile matter (page 14, Bulletin 107, section 4) be replaced by the "Determination of Total Organic Carbon" (J. Amer. Chem. Soc., 1904, 26: 1640).

In lieu of this it was recommended by Committee A that the method suggested for the determination of total organic carbon be referred to the committee on revision of methods for insertion as an additional determination, the original method to be also retained.

The recommendation of Committee A was adopted.

(6) INSECTICIDES.

It is recommended—

(1) That the Haywood-Davidson provisional method for determining total arsenic oxid in London purple be given a further trial, and that this be compared with the original provisional method. (Bul. 107, pp. 28, 29; also Circular 10.)

Adopted.

(2) That a further study of the amount of hydrochloric acid used in the reduction of London purple be made in order to determine whether 25 cc can be used instead of 50 cc.

Adopted.

(3) That the provisional precipitation method for the analysis of soda lye be made official. (Bul. 107, p. 31; also Circular 10.)

Referred to the referee for 1908 for final recommendation.

(4) That the provisional potassium acid sulphate method for the analysis of soda lye be dropped from the methods of the association.

Adopted.

(5) That the provisional hydrogen peroxid method for the determination of formaldehyde be made official. (Bul. 107, p. 33.)

Referred to the referee for 1908 for final recommendation.

(6) That the cyanid method for the determination of formaldehyde be further studied in order to determine more definitely what strength of formaldehyde may be estimated by this method. (Bul. 107, p. 33.)

Adopted.

(7) That the provisional hydrogen peroxid method for the determination of sulphur in sulphur dips be made official. (Bul. 107, p. 34.)

Referred to the referee for 1908 for final recommendation.

(8) That the methods proposed by J. K. Haywood for the analysis of lead arsenate (Proceedings 1906, Bul. 105, p. 165) be made provisional, and further studied with a view to their adoption by the association.

Adopted.

The association voted to hold the convention of 1908 in Washington and the meeting adjourned.

OBITUARY.

GEORGE CHAPMAN CALDWELL.
(1834-1907.)

WILBUR OLIN ATWATER.
(1844-1907.)

During the month of September, 1907, the death of two earnest and gifted workers in the field of scientific agriculture was reported, both chemists who worked with such enthusiasm and insight as to inspire all with whom they came in contact.

Dr. George Chapman Caldwell was for many years one of the leading writers and lecturers on agricultural experimentation, and the book "Agricultural Chemical Analysis," which he published in 1869, was the first of its kind and for a long time the English standard for agricultural science. He chose for his teachers both here and abroad the most famous scholars, and, as a consequence, gained a deep and broad knowledge of his subject. After having taught in several other colleges Doctor Caldwell was called to Cornell in 1868, being the first professor appointed. There he held the chair of chemistry for thirty-five years, during which time he served not only as teacher, but also as director of the experiment station of the university. In 1879, when the Cornell University Agricultural Experiment Station was organized, he was put at its head; and in 1887, when the station was reorganized, Doctor Caldwell was appointed chemist and retained this office until his retirement, twenty-five years later. While holding this directorship he published many valuable papers on the results of his work on fertilizers, cattle foods, sugar-beet culture, and methods of agricultural analysis.

He sought to inspire his students with the idea that thoroughness and accuracy were the fundamental principles of any work of lasting value. To his mind the possibilities for useful investigation in the field of agricultural science were unlimited, and he filled all who came in contact with him with equal enthusiasm, and in this way his ideals of work are perpetuated in his students and fellow-workers.

Dr. Wilbur Olin Atwater was an untiring laborer for the application of scientific methods to agriculture, and it was largely due to him that experiment stations were established in the United States.

When the first State experiment station was opened in Connecticut Doctor Atwater was appointed director. As other States realized the usefulness of this station and established similar ones, Doctor Atwater interested himself in them all, so that when the time came to demand national support he was fully equipped to represent the cause. In 1887, after the passage of the Hatch Act, the Storrs Agricultural Experiment Station was established, and Doctor Atwater was appointed its director. Much excellent work was done here in the twelve years during which he retained this directorship. Among the most interesting publications issued by the station was the continuation of Doctor

Atwater's studies on the absorption of atmospheric nitrogen by plants, studies which he had instituted as professor of chemistry at Wesleyan University.

When the work of the State experiment stations became so important that it was decided to establish an Office of Experiment Stations in the United States Department of Agriculture Doctor Atwater was summoned to take charge of it in addition to his other work. It was in this position that his wonderful executive ability had full play, for he aimed to make this national office a "clearing house and exchange for the State experiment stations." But with all his practical understanding Doctor Atwater was primarily a scientific investigator. His high ideals in this regard are expressed by his own statement: "The future usefulness of the stations will depend upon what they discover of permanent value, and this must come largely from the most abstract and profound research; to forget this will be fatal."

In 1889 he instituted the publication of a series of monographs on special subjects, under the name of Farmers' Bulletin, and these proved so popular that Congress appropriated a printing fund for them and arranged for their distribution on a large scale. The publication of the Experiment Station Record also was inaugurated by Doctor Atwater and proved of great value to agricultural scientists.

As the usefulness of the central office increased the work became too heavy for its director, who did not wish to relinquish entirely his work of teacher and investigator, and he therefore resigned, although he retained close relations with the office until the end of his active career.

Parallel with his studies in agricultural chemistry, Professor Atwater began early in his career to carry on investigations in physiological chemistry with special reference to human nutrition. In the early eighties, after a special study of problems and methods, he made the subject of human nutrition an important part of the work of the Connecticut Storrs Agricultural Experiment Station. Among the important results of this work may be mentioned the elaboration of experimental methods, the improvement of the bomb calorimeter, and the development of the respiration calorimeter in such form that the income and outgo of both matter and energy can be measured.

The accuracy of his scientific methods, the scope of his investigations, and his splendid understanding of practical conditions make his work valuable for all time. This, and the personality of Doctor Atwater, won the confidence of others, so that it was possible for him to carry out his ideas in successful practice. It was the rare combination in Doctor Atwater of a well-trained mind, splendid ability to organize and manage, and high scientific ideals that enabled him to be of such great service to humanity.

**OFFICERS, REFEREES, AND COMMITTEES OF THE ASSOCIATION OF
OFFICIAL AGRICULTURAL CHEMISTS FOR THE YEAR 1908.**

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Mr. HARRY SNYDER, St. Anthony Park, Minn.

Vice-President.

Mr. W. D. BIGELOW, Washington, D. C.

Secretary.

Mr. H. W. WILEY, Washington, D. C.

Additional members of the Executive Committee.

Mr. B. B. ROSS, Auburn, Ala.

Mr. G. S. FRAPS, College Station, Tex.

Referees.

Phosphoric acid: J. M. McCandless, Atlanta, Ga.

Nitrogen:

Determination of nitrogen: C. L. Penny, State College, Pa.

Separation of nitrogenous bodies: L. L. Van Slyke, Geneva, N. Y. (milk and cheese proteids).

Potash: B. B. Ross, Auburn, Ala.

Soils: S. D. Averitt, Lexington, Ky.

Dairy products: J. M. Bartlett, Orono, Me.

Foods and feeding stuffs: J. P. Street, New Haven, Conn.

Food adulteration: H. E. Barnard, Indianapolis, Ind.

Sugar: A. H. Bryan, Washington, D. C.

Tannin: F. P. Velch, Washington, D. C.

Insecticides: C. C. McDonnell, Washington, D. C.

Inorganic plant constituents: H. D. Haskins, Amherst, Mass.

Medicinal plants and drugs: L. F. Kebler, Washington, D. C.

Associate referees.

Phosphoric acid: W. F. Hand, Agricultural College, Miss.

Nitrogen:

Determination of nitrogen: G. W. Cavanaugh, Ithaca, N. Y.

Separation of nitrogenous bodies:

Meat proteids: P. F. Trowbridge, Columbia, Mo.

Vegetable proteids: E. B. Hart, Madison, Wis.

Potash: E. L. Baker, Geneva, N. Y.

Soils: J. G. Lipman, New Brunswick, N. J.

Dairy products: L. G. Michael, Ames, Iowa.

Foods and feeding stuffs: F. W. Morse, Durham, N. H.

Food adulterations:

Colors: H. M. Loomis, Galveston, Tex.

Saccharine products: Arthur Glven, Washington, D. C.

Fruit products: C. B. Cochran, West Chester, Pa.

Wine: Julius Horvet, St. Paul, Minn.

Beer: H. E. Barnard, Indianapolis, Ind.

Food adulterations—Continued.

Distilled liquors: L. M. Tolman, Washington, D. C.
 Vinegar: Chas. H. Illickey, Boston, Mass.
 Flavoring extracts: E. M. Chace, Washington, D. C.
 Spices: A. L. Winton, Chicago, Ill.
 Baking powder and baking chemicals: E. L. Redfern, Lincoln, Nebr.
 Meat and fish: F. C. Weber, Washington, D. C.
 Fats and oils: H. E. Bishop, Indianapolis, Ind.
 Dairy products: Herman Lythgoe, Boston, Mass.
 Cereal products: E. F. Ladd, Fargo, N. Dak.
 Vegetables: W. L. Dubois, Buffalo, N. Y.
 Condiments other than spices: E. M. Bailey, New Haven, Conn.
 Cocoa and cocoa products: A. G. Woodman, Boston, Mass.
 Tea and coffee: A. G. Woodman, Boston, Mass.
 Preservatives: W. D. Bigelow, Washington, D. C.
 Determination of water in foods: E. W. Brown, Washington, D. C.

Sugar:

Molasses methods: H. P. Agee, Audubon Park, La.
 Chemical methods: Fritz Zerban, Audubon Park, La.

Tannin: M. S. McDowell, State College, Pa.

Insecticides: Howard R. Watkins, Pullman, Wash.

Inorganic plant constituents: F. W. Robison, Lansing, Mich.

SPECIAL COMMITTEES.

Food Standards.

Mr. William Frear, State College, Pa., chairman.
 Mr. H. W. Wiley, Washington, D. C.
 Mr. H. A. Weber, Columbus, Ohio.
 Mr. M. A. Scovell, Lexington, Ky.
 Mr. E. H. Jenkins, New Haven, Conn.

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Revision of Methods.

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 Mr. J. H. Pettit, Urbana, Ill.
 Mr. F. W. Woll, Madison, Wis.

Committee to present the question of the unification of terms to the International Congress of Applied Chemistry.

- **Mr. R. J. Davidson, Blacksburg, Va., chairman.**
- Mr. C. G. Hopkins, Urbana, Ill.**
- Mr. W. D. Bigelow, Washington, D. C.**
- Mr. G. S. Fraps, College Station, Tex.**
- Mr. B. W. Kilgore, Raleigh, N. C.**
- Mr. H. J. Wheeler, Kingston, R. I.**
- Mr. J. T. Willard, Manhattan, Kans.**

CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This association shall be known as the Association of Official Agricultural Chemists of the United States. The objects of the association shall be (1) to secure uniformity and accuracy in the methods, results, and modes of statement of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership; and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to enter motions or vote in the association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. All persons eligible to membership shall become members ex officio and shall be allowed the privileges of membership at any meeting of the association after presenting proper credentials. All members of the association who lose their right to such membership by retiring from positions indicated as requisite for membership shall be entitled to become honorary members and to have all privileges of membership save the right to hold office and vote. All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote.

(3) The officers of the association shall consist of a president, a vice-president, and a secretary, who shall also act as treasurer; and these officers, together with two other members to be elected by the association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board whose members are eligible to membership, his office shall be considered vacant, and a successor may be appointed by the executive committee, to continue in office till the annual meeting next following.

(4) There shall be appointed by the executive committee, at the regular annual meeting, from among the members of the association, a referee and such associate referees for each of the subjects to be considered by the association as that committee may deem appropriate.

It shall be the duty of these referees to prepare and distribute samples and standard reagents to members of the association and others desiring the same, to furnish blanks for tabulating analyses, and to present at the annual meeting the results of work done, discussion thereof, and recommendations of methods to be followed.

(5) The special duties of the officers of the association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this association shall be held at such place as shall be decided by the association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

(7) No changes shall be made in the methods of analysis used in official inspection, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of the particular inspection affected to test the proposed changes.

(8) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(9) The executive committee will confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the association.

(10) All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by the approval of two-thirds of the members present entitled to vote.

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Issued September 12, 1908.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY—BULLETIN No. 117.
H. W. WILEY, Chief of Bureau.

COMMERCIAL SICILIAN SUMAC.

By

F. P. VEITCH,
CHIEF OF THE LEATHER AND PAPER LABORATORY.

INCLUDING NOTES ON THE MICROSCOPICAL EXAMINATION OF
SICILIAN SUMAC AND ITS ADULTERANTS,

By

B. J. HOWARD,
CHIEF OF THE MICROCHEMICAL LABORATORY.



WASHINGTON:
GOVERNMENT PRINTING OFFICE,
1908.

LETTER OF TRANSMITTAL.

UNITED STATES DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., June 3, 1908.

SIR: I have the honor to submit for your approval a report of two investigations, conducted in 1905 and 1907, concerning the quality of Sicilian sumac imported into this country, chemical and microscopical examination of a large number of samples having been made. This study was made in the Leather and Paper Laboratory of the Bureau of Chemistry because of its direct bearing on the leather trade interests of the country, as well as because of its relation to the increased production of sumac in the United States. Material assistance in the performance of the laboratory work involved in this investigation was rendered by H. H. Hurt and C. C. Smoot, of the Leather and Paper Laboratory. I recommend the publication of this report as Bulletin 117 of the Bureau of Chemistry

Respectfully,

H. W. WILEY,
Chief of Bureau.

Hon. JAMES WILSON,
Secretary of Agriculture.

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COMMERCIAL SICILIAN SUMAC.

QUANTITY AND VALUE OF IMPORTED SUMAC.

Sicilian sumac is the best vegetable tanning material known for pale colors and soft tannage, and is consequently extensively used for moroccos, roans, skivers, etc., and for brightening the color of leather tanned with dark materials. An extended investigation^a by a committee of the Society of Arts has shown conclusively that sumac-tanned leathers are less likely to be attacked by light and gas fumes, and hence better suited for use in bookbinding than any other known vegetable tannage.

As good "masculino," or Sicilian mountain sumac, contains from 25 to 35 per cent of tannin which is absorbed by hides, it is a very high grade and desirable tanning material, commanding a high price. Consequently it is adulterated to a considerable extent, and much complaint has arisen during the past three or four years both from importers and tanners about the mixing of sumac leaves with stems or other lower-grade and darker-colored substances, an adulteration which not only affects the material itself, but also darkens greatly the leather tanned. In view of these facts it has been deemed advisable to make a careful examination of imported Sicilian sumac.

Although mineral tanning has largely replaced vegetable tanning in the production of morocco, the importations of foreign sumac have remained about the same for a number of years, as shown by Table I, prepared from statistics issued by the Department of Commerce and Labor.

TABLE I.—Quantity and value of sumac imported into the United States from 1870 to 1907.

Year.	Quantity.	Value.
	<i>Pounds.</i>	<i>Dollars.</i>
1870.....	9,634,367	418,919
1875.....	16,718,678	511,941
1890.....	16,367,213	
1895.....	12,179,203	235,157
1900.....	10,335,980	
1905.....	15,583,334	225,036
1906.....	14,866,482	235,403
1907.....	12,244,907	259,974

^a Journal of the Society of Arts, London, 1901, p. 14.

CULTURE AND PREPARATION FOR MARKET.

Sicilian sumac (*Rhus coriaria*) is a shrubby bush which grows chiefly in Sicily and Tuscany, and succeeds on any well-drained soil, though the best development is secured in calcareous soils. That grown in the mountainous districts around Palermo is known as "masculino" and contains the highest percentage of tannin—from 25 to 35 per cent—while that grown on the plains is called "feminella" and usually contains less than 25 per cent. Andraesch^a states that "feminella" is a variety distinct from "masculino," stronger, having larger leaves, and containing a darker tannin but less of it than the "masculino." Examinations of both kinds of leaf have failed to show any differences, and communication with importers brings out the fact that no distinction is made except on a basis of the tannin content.

While in this country no attention is devoted to the cultivation of the native sumac, in Sicily it is commonly cultivated, as the yield and value of the leaf are both much greater than from the wild plant. Sumac may be grown on poor, stony, volcanic, or calcareous soils, not too far from the sea, and on mountain sides well exposed to the sun. Sudden changes of temperature or frequent rains, especially when the material is about ready to harvest, greatly injure its quality and strength.

The plant may be propagated from the young shoots which form each year about the mature plant, from cuttings of the well-ripened stem, or from the seed. The first method is the one generally followed. The shoots should be at least a foot high, be well supplied with buds, come from young, healthy plants, and have short chain roots well supplied with rootlets. When cuttings from the wood are made, they must be first rooted in a propagation frame at a temperature of about 70° F. The young plants are set in well-cultivated land in rows 2 feet apart, and are given three or four cultivations during the growing season to keep the land free from weeds and grass.

The first crop is harvested the year after planting, either by pruning or by picking the leaves. Harvesting begins about the middle of July, the time being governed by the development of the leaf, the object being to harvest when the leaf has acquired the deepest green color and reached its maximum weight. If the leaves are gathered by hand, harvesting begins when the first and lowest leaves have reached maturity, usually in May, and two subsequent gatherings are made as the younger leaves become fully developed, once late in July or early in August, and again in September, when the extremities of the branches are gathered. After being picked, the shoots and leaves

^a Abs., J. Soc. Chem. Ind., 1898, 17: 774.

are allowed to lie in the field in order that they may become partially cured, or they are immediately taken to the barn for curing. It is important that the material be not exposed to rains or to intense sunshine during curing, as the quality of the product is greatly injured thereby. As a rule, therefore, the best product may be obtained by drying under cover, being careful to turn the leaves frequently to prevent molding. After drying, the leaves and stems are roughly ground, baled, and sold as "leaf sumac," or they are reground in edge runner mills, sifted to remove the stems, ventilated, bagged, and sold as "ground sumac."

It can readily be seen that the dryness of the product and the proportion of stems that remains with the leaf of the baled and ground sumac will vary considerably according to the care with which it is handled. As these stems not only contain less tannin, but also have a deeper color than the leaf, the value of the product may be materially influenced simply by the method of preparation for market.

NATURE OF ADULTERATION.

In addition to the incorporation of large quantities of the stem with the leaf, a practice which must be regarded as an adulteration, a number of other materials less valuable for tanning than sumac are mixed with the leaf. By far the most common adulterant, indeed the one almost exclusively used in the sumacs imported into this country, is the leaf of *Pistacia lentiscus* commonly called lentiscus or lentisco. This leaf contains from 12 to 20 per cent of a catechol tannin, and leather tanned with sumac adulterated with this leaf darkens and reddens on exposure to air, for which reason its use is decidedly objectionable in the manufacture of certain grades of leather. The lentiscus is mixed with ground sumac at the rate of from 20 to 50 per cent, and with the sumac leaves at the rate of 20 to 30 per cent. Other leaves much less generally used in adulterating sumac are those of *Coriaria myrtifolia* ("stinco"), *Tamarix africana* ("brusca"), *Ailanthus glandulosa*, *Vitis vinifera* (grape vine), and some species of the *Rhus* family other than *coriaria*, as well as foreign material. Sumac from which tannin has been extracted or which has been injured by exposure is also mixed with the normal product. None of these adulterants can be detected by a casual examination of the sumac, but special methods, which will be described later, have been devised for this purpose.

The Italian laws require that all adulterated sumac offered for export shall be distinctly labeled with the kind and quantity of the adulterant, but it is claimed that this law is frequently evaded, and the trade journals state that a very large percentage of adulterated sumac has been shipped to this country. Some have tried to justify

this by asserting that the market here demands sumac at such a price that the genuine "masculino" sumac can not be sold. However this may be, it appears important that the actual conditions as to purity of imported sumac should be brought fully to the attention of the American importers and buyers, that they may take such steps as appear advisable for their protection.

INVESTIGATION OF 1905.

SECURING SAMPLES FOR ANALYSIS.

Through the cooperation of the customs division of the Treasury Department, the samples for examination were secured at the chief ports of entry in accordance with the instructions in the following letter:

JANUARY 17, 1905.

THE SECRETARY OF THE TREASURY.

SIR: In connection with the work of the Bureau of Chemistry of this Department it is desired to obtain samples of sumac leaves and ground sumac entering the ports of New York, Boston, and Baltimore. If in harmony with the regulations of your office, I should be glad if you would issue such instructions to the collectors of the ports named as will enable us to secure the material mentioned.

In taking the samples, the names and addresses of the consignor and of the consignee, together with a copy of all the marks on the bags, should be secured and these data forwarded with the proper samples.

Samples should be drawn from about 5 per cent of each invoice by passing a slotted sampling tube from top to bottom of the bags, thoroughly mixing the subsamples of each invoice thus obtained and taking from 1 to 2 pounds of this to be forwarded to this Department with the data above mentioned. * * *

In compliance with this request samples of leaf and ground sumac were taken by the Treasury Department, chiefly through the ports of New York and Boston, and delivered to the Bureau of Chemistry.

METHODS OF EXAMINATION.

The samples were submitted to both chemical and microscopic analysis in order to determine their composition and distinguish the adulterants if such were present. An extractor especially adapted to this purpose was used, numerous experiments^a having shown that with this apparatus the extraction of sumac is more complete and the operation is more easily conducted than with other extractors. Furthermore, the color of the resulting extract is less affected than when the ordinary copper Soxhlet, such as is quite commonly employed in tannery work, is used.

Inasmuch as the extraction is more complete with this form of extractor than with those formerly used, the results on tannin are,

^aJ. Amer. Chem. Soc., 1905, 27; 724; 1906, 28; 505.

as a rule, higher than those obtained and published a few years ago, when rarely more than 25 per cent of tannin was determined in even the best Sicilian sumac. The high results here reported, which are now obtained quite generally by others, are probably due, therefore, to more complete extraction and improved methods of analysis rather than to improvement in the quality of sumac. It has been the opinion among tannery chemists, based on the work of Semour-Jones, Palmer, Parker, and Proctor, of England, that most tanning materials are best extracted at temperatures below boiling; thus sumac is supposed to yield the highest results by extracting below 60° C. The work done in the Bureau of Chemistry makes this opinion no longer tenable, as the highest results have been obtained at from 60° to 90° C. according to the following method, which is now used in this laboratory in extracting all kinds of tannery materials.

Place in the extractor, preparatory to receiving the sample, a perforated porcelain disk and cover with a mat of asbestos or of purified cotton. Place the weighed sample of tanning material in a beaker and moisten with hot water at from 60° to 90° C. until it has the consistency of a thin paste; then transfer it to the extractor, removing all the material from the beaker with a jet of hot water. Let the water percolate through the extractor into a Jena boiling flask, press the material down well, cover with a perforated porcelain plate, and return the percolate to the extractor until it runs clear. Allow a total volume of from 300 to 400 cc to percolate at a temperature of from 60° to 90° C. Place about 250 cc of fresh water in a clean receiving flask and connect it with the extractor by means of a block-tin condenser, heat to boiling, and finish the extraction at steam heat, replacing the extract with fresh portions of water two or three times and being careful to keep the total volume of extract within a liter. When the extraction is completed, usually in from twenty to twenty-five hours, combine the hot extracts in a liter flask and make up to volume when cold. Only the best nonsoluble glass and block tin must be used in the extraction apparatus, as the alkali dissolved from ordinary glass materially dissolves "reds" insoluble in cold water. Make the determination of tannin and other constituents of the extracts according to the official methods of the Association of Official Agricultural Chemists.^a

Determine the moisture by drying 5 grams of substance in a flat-bottomed dish for five hours and check the weight again after drying for three hours.

Determine the crude ash in the residue from the moisture by incineration at a low red heat until all carbon is burned away, then cool and weigh the residue.

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 107, p. 35.

To determine the sand, treat the ash with about 10 per cent hydrochloric acid and warm gently for several hours, filter, wash thoroughly, ignite, and weigh. The weight so obtained is considered as sand.

Make the color determination of the extract with the Lovibond tintometer, the readings being made in the 1-inch cell on the soluble solids filtrate, and calculate the results to a basis of 0.5 per cent of tannin in the solution. Give the results in terms of red and yellow, the black being subtracted for the red and yellow readings.

It should be borne in mind that the color determination so obtained does not necessarily represent the color of the extract obtained in tannery practice. Indeed, it is almost certain that the color of the extracts as thus prepared for analysis is considerably deeper than that of the extract obtained from the same materials by ordinary tannery methods. These results, then, only show the relative colors produced by different samples under like conditions of extraction and should be compared only among themselves.

DISCUSSION OF RESULTS OF ANALYSIS.

In tabulating the data on these samples, the name and address of the consignor, when these appeared on the containers, the name and address of the consignee, the approximate date of entry, and the place of sampling are given in connection with the chemical analysis and the microscopic examination. Care was taken to secure samples only from invoices imported in good condition.

As is shown in Table II (p. 12), the average percentage of tannin in all the samples of sumac is 28.8 per cent, which is higher than the results generally given in the literature for Sicilian sumac. As has been said, it is not believed that this is due to any improvement in the character of the leaf now grown, but rather to improved methods of extraction and also to changes in methods of analysis.

Approximately 41 per cent of the invoices from which samples were taken were mixed with lentiscus, this being practically the only adulterant employed, except sumac stems, which were present in excessive quantities in a number of samples. The adulterated samples contained from 19.6 per cent to 33.3 per cent and averaged 26.6 per cent of tannin, or 2.2 per cent less than the average of all the sumac samples. A number of the samples contained an excess of sumac stems, and the average tannin content of these was 29.9 per cent, which indicates that the stems are not added in such large quantities as is the lentiscus. The samples of pure sumac contained from 27.4 to 35.1 per cent and averaged 31.9 per cent of tannin.

Adulteration of the average pure sumac with 30 per cent of the average lentiscus would yield an article having the same percentage

of tannin as the average adulterated samples—that is, approximately 27 per cent. The figures, therefore, indicate an average adulteration of 30 per cent of lentiscus, which is possibly below the actual practice, as high-grade sumacs are more likely to be adulterated than the lower grades. Several of the samples, however, contain so little tannin that it is evident that either an exceptionally low grade of sumac was used or that lentiscus was almost entirely substituted.

Ten samples were marked as containing lentiscus. In two cases the examination proved the sample to be pure sumac. In no case was more than a 25 per cent adulteration admitted. The average tannin content of the six admittedly adulterated samples is 27.5 per cent, practically identical with the average of all the adulterated samples.

If the general statement that the “feminella” sumac contains less than 25 per cent of tannin is accepted, it would appear that none of the adulterated samples was plain-grown sumac. As this classification seems to be based solely on the tannin content, it may be ignored except in so far as it is an expression of the agricultural fact that sumac grown on the high ground contains more tannin than that grown in the valleys.

Leather tanned with sumacs adulterated with lentiscus is darker than that tanned with pure Sicilian sumac, and the determination of the color of the extracts from these samples is in harmony with this fact. The darkest extract from a pure sumac contains less red coloring matter than the lightest-colored extract from an adulterated sample, while the extracts from the samples of lentiscus contained several times as much coloring matter as the darkest pure sumac extract.

TABLE II.—*Chemical and microscopical examination of Sicilian sumac sampled in 1905.*

Laboratory No.	Consignor.	Consignee.	Collection of samples.		Statement on bags as to purity.	Chemical examination (per cent).										Microscopical examination.	Presence of lentiscus as indicated by color after drying.
			Place.	Date.		Molature.	Ash.	Band.	Total extract.	Soluble extract.	Insoluble extract.	Non-tannins.	Available tannins.	Red.	Yellow.		
74	G. S. Benanti, Palermo.	H. M. Rau, New York.	Boston.		Ground sumac.	8.06	7.57	1.62	50.04	4.6	5.4	18.62	0.3	3.8	6.7	Lenticus abundant.	Lenticus.
75	Giovanni Riso, Palermo.	O. S. Janney & Co., Boston.	do.		do.	8.05	7.65	1.44	40.46	4.5	4.1	20.12	4.4	6.10	7.7	do.	Do.
76	P. Savona & Co., Palermo.	A. Klipstein, Boston.	do.		do.	8.08	7.50	1.46	40.47	0.0	2.9	19.87	2.2	3.8	9.4	do.	Do.
77	do.	do.	do.		do.	8.54	6.99	1.33	51.04	4.3	2.7	19.42	8.9	3.5	8.7	do.	Sumac.
78	G. Terrasi, Palermo.	Windsor Bros. & Smith, Boston.	do.	Jan. 31, 1905	do.	9.00	8.40	1.55	55.25	1.8	3.4	21.08	0.8	3.2	12.8	Pure sumac.	Do.
79	Unknown.	John Harper & Co., Philadelphia.	Philadelphia.		Pure leaf sumac.	7.42	6.17	.38	58.75	4.4	3.3	20.33	5.1	1.3	3.8	do.	Do.
80	do.	do.	do.		15 per cent lentiscus leaf	7.88	6.29	.48	58.05	2.2	2.8	19.93	3.3	1.9	5.6	Some lenticus.	Do.
81	do.	do.	do.		25 per cent lentiscus leaf	7.73	6.12	.91	53.81	8.8	0.20	9.30	9.9	2.9	6.4	do.	Do.
82	G. B. Casiglia & Figlio, Palermo.	J. S. Bent, Boston.	Boston.		Ground sumac.	8.01	9.19	16.35	1.37	47.1	4.2	18.72	4.3	6.10	5.5	do.	Do.
83	M. Polero & Co., Palermo.	W. L. Montgomery & Co., Boston.	do.		Pure Sicily sumac leaf	6.93	7.60	.96	55.35	1.5	3.8	19.32	2.2	1.7	6.5	Stems rather abundant.	Do.
85	G. Dalla & Fi., Palermo.	W. D. Byron & Sons, Williamsport, Md.	Williamsport, Md.		Superior quality warranted pure.	8.66	8.36	.98	55.51	3.3	4.2	18.73	2.6	2.1	7.2	Pure sumac.	Do.
86	P. Savona & Co., Palermo.	do.	do.		Guaranteed pure extra ventilated.	8.29	7.45	1.18	52.24	7.5	4.7	19.42	8.1	4.9	8.9	Lenticus abundant.	Lenticus.
87	M. Polero & Co., Palermo.	do.	do.		Warranted genuine prime quality.	8.21	8.09	1.11	55.95	1.6	4.3	19.83	1.8	2.3	6.3	Some stems.	Sumac.
88	G. Dalla & Fi., Palermo.	W. L. Montgomery & Co., Boston.	Boston.		Pure ground sumac.	7.15	8.45	1.39	56.52	1.1	4.4	19.52	2.6	2.5	6.7	Pure sumac.	Do.
89	Unknown.	J. M. Harper & Co., Philadelphia.	Philadelphia.		Powdered lentiscus.	6.70	6.74	1.45	45.03	4.4	5.6	22.21	7.2	10.1	14.1	Lenticus only.	
90	Giovanni Terrasi, Palermo.	do.	do.		Pure ground sumac.	6.81	9.21	1.68	56.62	5.4	4.1	19.63	2.9	1.7	6.0	Sumac only.	Sumac.

124	Francesco Basso & Co., Palermo.	J. S. Young & Co., Baltimore.	Guaranteed pure leaf, extra ventilated.	7.61	6.91	73.62	440.1	3.320	3.28.8	2.9	7.9	Lentiscus abundant, some stems.	Do.
125	G. S. Benanti, Palermo.	do.	Warranted pure leaf, extra ventilated.	8.13	7.50	94.50	146.9	3.218	728.2	2.2	6.7	Some sumac stems	Do.
126	Francesco Basso & Co., Palermo.	do.	Guaranteed pure leaf.	6.80	6.11	62.57	354.8	2.521	533.3	2.3	6.0	Pure sumac.	Do.
127	G. S. Benanti, Palermo.	do.	Warranted pure leaf, extra ventilated.	7.00	6.36	44.62	540.7	2.820	620.1	2.3	7.3	Some stems	Do.
128	Francesco Basso & Co., Palermo.	do.	do.	7.46		52.74	6	3.120	029.6	2.1	7.7	Lentiscus abundant.	
129	P. Savona & Co., Palermo.	Pfister & Vogel, Milwaukee.	do.	6.67	8.54	1.63	54.048.1	5.920	128.0	9.327.8		Lentiscus very abundant.	Lentiscus.
132	Unknown.	Stamford Mfg. Co., Stamford, Conn.	Powdered lentiscus.	8.07	8.84	1.46	45.030.1	5.922	716.4	7.837.4		Lentiscus only.	Do.
133	do.	do.	Ground lentiscus.	8.17	6.24	78.42	237.8	4.420	617.2	9.721.9		Lentiscus and some stems.	Do.
135	Giovanni Riso, Palermo.	O. S. Janney & Co., New York.	Ground sumac.	6.93	8.02	1.36	50.144.6	5.519	125.5	5.015.4		Lentiscus abundant, trace of tamarix.	Do.
136	Pro forma entry.	F. R. Leonori & Co., New York.	100 per cent warranted pure, extra ventilated.	7.50	9.20	1.50	57.051.1	5.919	032.1	2.3	9.4	Pure sumac.	Sumac.
137	Giovanni Riso, Palermo.	Brown Bros. & Co., New York.	Guaranteed pure.	7.11	7.78	1.28	48.844.4	4.418	625.8	4.516.5		Lentiscus abundant.	Lentiscus.
138	Giovanni Terrasi, Palermo.	Harbler & Co., New York.	100 per cent warranted pure, extra ventilated.	7.44	8.46	1.51	56.151.1	5.018	632.5	1.9	9.0	Lentiscus, trace.	Sumac.
139	Paolo Graziano, Palermo.	D. A. Shaw & Co., New York.	Superior quality, warranted pure.	7.01	8.37	1.84	50.046.2	3.820	825.4	4.015.5		Lentiscus abundant.	Lentiscus.
140	P. Mormino, Termini.	B. Volgt, New York.	Superior quality, warranted pure.	6.98	8.51	1.63	50.246.0	4.218	627.4	3.611.8		do.	Do.
141	Giovanni Riso, Palermo.	Core & Herbert, New York.	Superior quality, warranted pure.	7.32	7.32	1.14	48.944.2	4.718	625.3	4.614.8		do.	Do.
143	Paolo Graziano, Palermo.	Winter & Smille, New York.	Guaranteed pure, extra ventilated.	7.22	6.53	1.29	45.542.4	3.122	819.6	11.223.4		do.	Do.
144	P. Savona & Co., Palermo.	A. Klipstein, New York.	Superior quality, warranted pure.	6.34	8.00	79.51	646.8	4.821	125.7	2.3	7.5	Some stems	Sumac.
147	G. Dalla & Fi., Palermo.	W. L. Montgomery & Co., New York.	Ground sumac, guaranteed pure, prime quality.	7.36	8.70	1.65	64.751.2	3.520	131.1	2.3	8.5	Pure sumac.	Do.
148	M. Pojero, Palermo.	W. L. Montgomery & Co., Boston.	Extra ventilated, 100 per cent pure.	7.24	8.78	1.62	55.151.4	3.718	632.8	1.4	5.1	do.	Do.
149	E. Bertini, Palermo.	Winslow Bros. & Smith Co., Boston	do.	7.02	8.81	1.73	56.652.6	4.017	734.9	1.6	5.9	do.	Do.

TABLE II.—*Chemical and microscopical examination of Sicilian sumac sampled in 1905—Continued.*

Laboratory No.	Consignor.	Consignee.	Collection of samples.		Statement on bags as to purity.	Chemical examination (per cent).								Microscopical examination.	Presence of lentiscus as indicated by color after drying.		
			Place.	Date.		Moisture.	Ash.	Sand.	Total extract.	Soluble extract.	Insoluble extract.	Non-tannins.	Available tannins.			Color in one-half per cent solution.	
																Red.	Yellow.
150	P. Savona & Co., Palermo.	A. Klipstein & Co., Boston.	Boston.	Mar. 17, 1905	Guaranteed pure, extra ventilated.	6.90	9.12	1.90	48.5	45.4	3.1	18.6	24.8	4.0	11.7	Some lentiscus.	Lentiscus.
151	do.	do.	do.	do.	do.	7.15	8.59	1.78	49.2	46.3	2.9	18.5	27.8	4.1	9.8	do.	Do.
152	Fratelli Savona, Palermo.	W. L. Montgomery & Co., Boston.	do.	Mar. 20, 1905	Extra ventilated, warranted pure.	7.15	9.66	2.49	53.9	47.6	6.3	20.5	27.1	3.6	17.9	do.	Do.
153	V. Vitruano, Palermo.	Herfzel, Feltmann & Co., New York.	do.	do.	Pure sumac.	7.61	8.95	1.58	57.2	50.5	6.7	20.2	30.3	2.3	10.4	Pure sumac.	Sumac.
154	E. Bertini, Palermo.	A. C. Lawrence, Leather Co., Boston.	do.	Mar. 18, 1905	Extra ventilated, guaranteed pure.	7.54	7.88	1.55	57.5	53.0	4.5	19.0	34.0	.8	4.7	do.	Do.
155	Giovanni Riso, Palermo.	O. S. Janney & Co., Boston.	do.	do.	Ventilated, warranted pure.	7.78	8.07	1.34	52.5	48.7	3.8	19.1	29.6	2.2	10.9	Some lentiscus.	Do.
156	C. Wederkind Co., Palermo.	Leber & Meyer, New York.	do.	Mar. 20, 1905	Extra ventilated.	7.33	7.42	1.24	50.0	46.7	3.3	19.8	26.9	4.0	12.4	Lentiscus abundant.	Lentiscus.
157	V. Vitruano di Gipe, Palermo.	Herfzel, Feltmann & Co., New York.	do.	do.	Pure, ventilated.	7.52	9.56	1.99	54.5	49.4	5.1	19.6	29.8	2.4	11.6	Pure sumac.	Sumac.
158	Salvatore Terrasi, Palermo.	E. F. Mulholland Co., Boston.	do.	Mar. 23, 1905	Ventilated, guaranteed pure.	7.53	9.13	2.17	54.8	50.2	4.6	17.5	32.7	1.6	7.3	do.	Do.
177	Giovanni Terrasi, Palermo.	Gillespie Bros. & Co., New York.	New York	Apr. 7, 1905	Warranted pure, ventilated.	7.37	9.91	2.48	55.7	50.6	5.1	19.0	31.6	1.8	10.2	do.	Do.
178	Unknown.	A. C. Lawrence, Leather Co., Boston.	Boston.	Apr. 13, 1905	Guaranteed pure.	7.10	8.67	1.50	57.4	53.2	4.2	20.1	33.1	1.6	7.3	do.	Do.
179	P. Savona, Palermo.	A. Klipstein & Co., Boston.	do.	do.	do.	7.50	8.19	1.40	49.7	46.2	3.5	18.6	27.6	3.7	11.5	Lentiscus abundant.	Lentiscus.
180	do.	do.	do.	do.	do.	7.56	8.49	1.33	50.3	47.0	3.3	20.7	26.3	3.5	15.6	do.	Do.
181	C. D'Arice & filie, Palermo.	G. W. Varney Co., Boston.	do.	do.	Warranted 100 per cent pure.	7.46	8.79	1.51								Some lentiscus.	Do.
182	Office of Botanical Investigations, U. S. Dept. Agr.	Washington, D. C.	Washington, D. C.	do.	Lentiscus.	8.52	6.40	.42	40.7	37.8	2.9	24.0	13.8	5.8	15.0	Lentiscus only.	Lentiscus.
183	P. Polo Graziano, Palermo.	do.	do.	do.	do.	6.47	5.98	.41	42.4	39.3	3.1	25.2	14.1	6.8	15.9	do.	Do.
193	do.	D. A. Shaw & Co., New York.	New York	Mar. 6, 1905	do.	6.99	9.69	2.28	46.7	41.8	4.9	18.4	23.4	4.4	13.5	Lentiscus abundant.	Do.

194	Unknown.	H. M. Rau, New York.	do.	Apr. 20, 1905	Warranted pure leaf.	6.00	10.10	2.28	51.7	48.4	3.320	228.2	2.812	1.	Pure sumac.	Sumac.
195	Unknown.	do.	do.	Apr. 20, 1905	Pure leaf.	6.77	7.20	9.65	4.850	4.4	4.418	0.82	4.1	6.7	do.	do.
196	do.	do.	do.	do.	Warranted pure.	3.49	10.53	2.55	0.9	43.6	3.419	227.4	3.811	5.	do.	do.
197	Shumac Steam Mills, Termini.	B. Volgt, New York.	do.	do.	25 per cent lentiscus warranted.	7.46	8.74	1.89	46.7	46.0	3.719	0.27	3.0	9.7	Lentiscus abundant, some tamari.	1
198	C. Wederkind & Co., Palermo	Leber & Sons, New York.	do.	Apr. 18, 1905	Pure.	6.88	7.47	1.35	50.4	45.9	4.519	3.26	6.		Lentiscus abundant.	do.
199	Unknown.	do.	do.	do.	20 percent lentiscus, extra ventilated.	7.39	8.79	1.89	53.3	49.2	4.118	830.4			Sumac only.	do.
200	do.	O. S. Janney & Co., New York.	do.	do.	Warranted pure.	7.48	7.94	1.94	48.6	43.2	5.419	0.24	2.4	512.1	Lentiscus abundant.	do.
201	Fratelli Savona, Palermo.	W. L. Montgomery & Co., Boston.	do.	Mar. 6, 1905		9.17	9.95	2.44	40.0	44.9	4.118	926.0			Some lentiscus.	do.
202	G. Dalla & Fi., Palermo.	do.	do.	do.		7.24	8.63	1.24	54.3	50.0	4.319	130.9			Pure sumac.	do.
218	Salvatore Terrasi, Palermo.	A. B. Clark Co., Boston.	Boston.	May 2, 1905	Guaranteed pure.	7.67	8.29	1.73	57.6	52.1	5.518	733.4	1.8	6.2	do.	do.
312	Unknown.	O. S. Janney & Co., New York.	New York.	Apr. 29, 1905	Warranted pure.	7.32	8.27	1.42	47.2	41.7	5.519	921.8	6.015	4.	Much lentiscus, some tamari.	do.
313	do.	do.	do.	do.	do.	7.52	7.51	1.15	48.3	43.1	5.219	723.4	4.712	6	Lentiscus abundant.	do.
322	do.	do.	do.	do.												do.
323	do.	do.	do.	do.												do.
324	do.	H. M. Rau, New York.	New York.	May 31, 1905	Ventilated, pure.	8.07	8.66	1.94	54.7	49.5	5.218	131.4	1.9	7.9	Pure sumac.	do.
325	Fratelli Savona, Palermo.	W. L. Montgomery & Co., New York.	do.	do.	Warranted pure, extra ventilated.	8.00	10.01	3.05	40.2	45.0	4.217	927.1	3.812	1	Lentiscus abundant.	do.
326	C. Wederkind & Co., Palermo.	Leber & Son, New York.	do.	do.	Pure, extra ventilated.	8.09	8.39	1.70	55.0	50.6	4.420	130.5	3.310	6	Sumac only.	do.
327	F. G. Pipitone, Palermo.	Plaster & Vogel Leather Works, New York.	do.	May 31, 1905	Warranted extra ventilated.	8.00	8.77	2.07	46.1	41.7	4.418	423.3	5.717	3	Lentiscus abundant.	do.
328	Unknown.	A. Kilpstein & Co., New York.	do.	do.	Pure, ventilated.	8.06	9.42	2.08	50.9	46.3	4.617	129.2	2.8	8.9	Sumac only.	do.
329	do.	do.	do.	do.	Pure sumac.	8.04	8.57	1.97	57.4	52.8	4.619	633.2	2.4	7.8	do.	do.
330	P. Savona & Co., Palermo.	do.	do.	June 5, 1905	Guaranteed pure.	7.96	8.16	1.92	48.3	44.2	4.117	926.3	3.5	9.9	Lentiscus abundant.	do.
331	Unknown.	do.	do.	do.	10 percent lentiscus, ventilated.	7.93	9.61	2.08	53.9	49.2	4.719	529.7	3.1	9.8	Lentiscus abundant, possibly some tamari.	do.
332	Mormino & Fi., Termini.	B. Volgt, New York.	do.	May 31, 1905	25 percent lentiscus, Bull brand.						3.318	826.9	4.411	0	Lentiscus abundant.	do.
334	Salvatore Terrasi, Palermo.	A. B. Clark Co., Boston.	Boston.	May 15, 1905	Warranted pure.	8.16	8.30	1.59	59.2	54.0	5.219	234.8	2.4	7.9	Sumac only.	do.
335	M. Pojaro, Palermo.	W. L. Montgomery & Co., Boston.	do.	June 28, 1905	Guaranteed pure, warranted genuine.	8.26	8.29	1.27	56.8	51.4	5.420	231.2	2.5	9.4	do.	do.
336	do.	do.	do.	do.	Guaranteed pure.	7.78	8.26	34	55.2	51.7	4.520	231.5	2.4	9.3	do.	do.

TABLE II.—*Chemical and microscopical examination of Sicilian sumac sampled in 1905—Continued.*

Laboratory No.	Consignor.	Consignee.	Collection of samples.		Statement on bags as to purity.	Chemical examination (per cent.).								Microscopical examination.	Presence of lentiscus as indicated by color after drying.
			Place.	Date.		Moisture.	Ash.	Sand.	Total extract.	Soluble extract.	Insoluble extract.	Non-tannins.	Available tannins.	Color in one-half per cent solution.	
														Red.	Yellow.
337	E. Bertini, Palermo.	Windsor Bros. & Smith Co., Boston.	Boston.	June 28, 1905	Warranted 100 per cent pure, extra ventilated.	7.97	7.67	1.56	58.653.8	4.818.934.9	1.7	6.8	Sumac only.		
338	P. Savona & Co., Palermo.	A. Kilpstein & Co., Boston.	do.	do.	Guaranteed pure, extra ventilated.	8.12	8.09	1.34	57.951.2	6.719.731.5	2.8	10.0	Lentiscus, small amount.		
339	Unknown.	O. S. Janney & Co., Boston.	do.	June 2, 1905	Warranted pure, extra ventilated.	7.86	8.83	1.53	54.840.4	5.420.028.4	3.0	11.8	Possibly an excess of stems.		
340	E. & A. Graziano, Palermo.	do.	do.	May 12, 1905	do.	7.40	8.62	1.56	53.440.2	4.220.028.2			Sumac only.		
341	G. Dalia & Fl., Palermo.	W. L. Montgomery & Co., Boston.	do.	June 2, 1905	Warranted pure.	8.44	8.53	1.38	54.950.0	4.919.230.8	2.0	8.5	do.		
342	S. Fuglisi, Palermo.	do.	do.	do.	25 per cent lentiscus, ventilated.	7.69	8.91	1.62	46.444.8	4.617.926.9	3.5	11.0	Lentiscus abundant.		
343	G. B. Cadigia & Figlio, Palermo.	J. S. Bent, Boston.	do.	May 15, 1905	Strong and pure, extra ventilated.	7.61	9.56	1.83	52.747.4	5.317.430.0	2.0	8.0	Sumac only.		
344	E. Bertini, Palermo.	Windsor Bros. & Smith Co., Boston.	do.	June 6, 1905	Warranted 100 per cent pure, extra ventilated.	8.14	7.87	1.70	57.452.9	4.518.434.5	3.5	10.0	do.		
345	E. & A. Graziano, Palermo.	O. S. Janney & Co., Boston.	do.	May 12, 1905	Warranted pure.	7.38	7.41	1.34	50.945.6	5.320.025.6	2.2	7.1	Lentiscus abundant, possibly some tamarix.		
346	E. Bertini, Palermo.	O. L. Lawrence Leather Co., Boston.	do.	June 6, 1905	Guaranteed pure, extra ventilated.	8.20	7.88	1.51	56.552.6	3.918.733.9	1.3	4.7	Sumac only.		
347	do.	do.	do.	do.	do.	7.90	7.46	1.11	57.252.7	4.518.134.6	2.8	7.5	do.		
348	Fratelli Savona, Palermo.	W. L. Montgomery & Co., Boston.	do.	May 12, 1905	Warranted pure, extra ventilated.	7.37	9.23	2.01	62.648.5	4.119.426.1	3.4	10.1	Lentiscus abundant.		
349	P. Savona, Palermo.	A. Kilpstein & Co., Boston.	do.	do.	Guaranteed pure, extra ventilated.	7.46	8.26	1.41	46.245.5	3.719.126.4	3.7	11.1	do.		
350	do.	A. Kilpstein & Co., New York.	New York.	June 6, 1905	Guaranteed pure.	8.06	8.08	1.40	48.345.1	3.218.726.4	3.5	11.1	do.		
351	do.	do.	do.	May 8, 1905	Guaranteed pure, extra ventilated.	7.92	7.99	1.33	46.245.6	3.619.925.7	4.5	11.5	Lentiscus very abundant.		

332	E. & A. Graziano, Palermo.	do.	May 2, 1905	Warranted pure.	7.95	7.14	1.22	45.5	41.0	4.520	720.3	8.717.1	do.	
333	Salvatore Terrasi, Palermo.	do.	June 30, 1905	Guaranteed pure, ventilated.	8.05	8.42	1.34	54.1	50.8	3.318	232.6	1.8	7.0	Barely a trace of lentiscus.
334	Unknown.	do.	June 13, 1905	do.	8.08	9.40	2.42	48.9	45.6	3.318	027.6	3.212.8	Sumac only.	Sumac.
335	Salvatore Terrasi, Palermo.	do.	June 23, 1905	Guaranteed pure.	7.76	8.40	1.71	56.0	51.6	4.416	934.7	1.2	6.5	do.
336	C. Wederkind & Co., Palermo.	do.	May 8, 1905	Pure, extra ventila- ted.	7.70	9.00	2.42	51.9	48.4	3.517	431.0	2.4	7.3	do.
337	Giovanni Terrasi, Palermo.	do.	July 6, 1905	Warranted 100 per cent pure.	9.46	8.18	1.28	54.3	50.1	4.216	633.5	1.8	7.8	do.
338	do.	do.	June 10, 1905	do.	7.98	7.58	1.33	55.7	51.3	4.417	134.2	1.8	7.6	do.
339	do.	do.	July 15, 1905	Guaranteed pure.	8.47	8.53	1.99	54.2	50.3	3.918	831.5	1.8	8.0	do.
340	do.	do.	June 2, 1905	Warranted pure.	7.84	9.13	1.27	53.8	50.2	3.619	630.6	2.3	6.8	do.
341	G. Dalla & Fi., Pa- lermo.	do.		Guaranteed pure.	7.65	7.87	1.22	46.8	46.7	3.119	327.4	3.7	9.6	Lentiscus abun- dant.
342	P. Savona & Co., Palermo.	do.	June 29, 1905	do.	7.65	9.23	1.53	51.6	48.5	3.119	026.5	2.6	8.2	Sumac only.
343	Francisco Basso, Palermo.	do.	July 12, 1905	25 per cent lentis- cus, warranted pure sumac.	7.83	8.09	1.44	56.6	52.6	4.019	233.4	2.0	6.2	do.
344	Unknown.	do.		Warranted pure.	7.84	9.02	1.25	54.6	50.9	3.719	931.0	1.8	6.4	do.
345	Fratelli Verga, Pa- lermo.	do.	July 27, 1905	do.	7.78	9.00	1.41	55.0	50.9	4.120	130.8	1.8	7.4	do.
346	Unknown.	do.	June 30, 1905	25 per cent lentis- cus.	7.50	8.22	1.69	51.4	48.0	3.418	326.7	3.4	9.9	Lentiscus abun- dant.
347	C. Wederkind & Co., Palermo.	do.	June 24, 1905	Pure sumac.	7.70	8.68	1.44	54.7	50.8	3.919	831.0	1.8	8.4	Sumac only.
348	P. Savona & Co., Palermo.	do.		do.	7.63	7.65	1.31	46.7	46.2	3.518	927.3	3.712.5	Lentiscus abun- dant.	Lentiscus.
349	Unknown.	do.	July 18, 1905	Guaranteed pure.	7.72	8.90	1.59	56.6	51.2	5.419	431.8	2.3	8.4	Sumac only.
350	E. Bertini, Palermo.	Boston.	Aug. 3, 1905	Guaranteed pure, extra ventilated.	8.04	8.52	1.44	50.9	47.6	3.316	531.1		do.	do.
351	do.	do.	July 31, 1905	do.	8.46	7.56	1.16	55.8	51.2	4.617	433.8		do.	do.
352	C. Wederkind & Co., Palermo.	New York.	June 8, 1905	Pure.	7.77	9.04	1.60	53.1	48.2	4.918	326.9		do.	do.
353	do.	do.	May 11, 1905	Warranted pure.	7.77	8.69	1.46	54.8	50.0	4.818	731.3	1.4	10.4	do.
354	G. Sansone, Palermo	do.	July 23, 1905	extra ventilated.	8.68	8.26	1.54	52.8	48.1	3.718	730.4	1.7	7.6	Lentiscus, small amount.
355	G. Dalla & Fi., Pa- lermo.	do.		Warranted pure.	8.49	8.29	1.37	53.4	50.4	3.019	231.2	1.7	8.3	Sumac only.
356	Unknown.	do.	Apr. 24, 1905	do.	7.79	7.47	1.29	46.8	44.1	3.720	025.1	4.5	9.8	Lentiscus abun- dant.
357	Giovanni Terrasi, Palermo.	do.	May 8, 1905	100 per cent guar- anteed pure, ex- tra ventilated.	8.20	7.76	1.28	53.4	53.3	5.119	034.3	1.4	6.7	Sumac only.

TABLE II.—*Chemical and microscopical examination of Sicilian sumac sampled in 1905—Continued.*

Laboratory No.	Consignor.	Consignee.	Collection of samples.		Statement on bags as to purity.	Chemical examination (per cent).										Microscopical examination.	Presence of lentiscus as indicated by color after drying.
			Place.	Date.		Moisture.	Ash.	Sand.	Total extract.	Soluble extract.	Insoluble extract.	Non-tannins.	Available tannins.	Color in one-half per cent solution.			
														Red.	Yellow.		
400	P. Savona & Co., Palermo.	A. Klipstein & Co., New York.	New York	May —, 1905	Guaranteed pure.	8.09	7.43	1.06	48.8	46.1	2.7	19.6	24.5	4.1	11.2	Lentiscus abundant.	Lentiscus.
401	Dott. F. Niceta, Palermo.	Zinsser & Co., Inc., Hastings-on-Hudson.	do.	June 23, 1905	Pure leaf.	7.95	7.93	.50	56.6	53.0	3.6	19.8	33.2	1.7	5.2	Sumac only.	Sumac.
402	Unknown.	H. M. Rau, New York.	do.	June 29, 1905	Finest quality, ventilated.	7.62	8.43	1.05	55.4	51.3	4.1	19.1	32.2	1.5	7.2	do.	Doubtful.
403	Mormino & Fi., Palermo.	B. Volgt, New York.	do.	June 14, 1905	25 per cent lentiscus, ventilated, warranted.	7.77	10.95	3.05	47.3	44.4	2.9	19.4	25.0	4.8	14.2	Lentiscus abundant.	Lentiscus.
404	Giovanni Terrasi, Palermo.	F. R. Leonori & Co., New York.	do.	July 29, 1905	100 per cent warranted, pure Sicily sumac.	8.03	8.58	1.30	55.2	51.4	3.8	18.8	32.6	1.5	8.0	Sumac only.	Sumac.
407	Francisco Basso & Co., Palermo.	H. M. Rau, New York.	do.	May 22, 1905	Guaranteed pure, extra ventilated.	7.15	7.46	.68	50.9	48.0	2.9	20.4	27.6	3.7	9.7	Some lentiscus.	Lentiscus.
408	Unknown.	do.	do.	July 27, 1905	Pure leaf, first quality.	7.40	7.22	.69	55.5	51.6	3.9	20.5	31.1	2.7	7.3	Sumac only.	Do.
409	Dott. F. Niceta, Palermo.	Zinsser & Co., Inc., Hastings-on-Hudson.	do.	May 31, 1905	Pure leaf.	7.89	7.66	.60	57.2	53.9	3.3	19.6	34.3	1.8	5.2	do.	Sumac.
410	do.	do.	do.	June 10, 1905	do.	8.18	7.85	.90	56.0	52.5	3.5	20.0	32.5	1.8	6.4	do.	Do.
411	V. Vitano, Palermo.	H. M. Rau, New York.	Baltimore.	do.	Sicily leaf.	7.82	6.40	.38	55.5	52.4	3.1	20.0	32.4	1.3	4.9	do.	Do.
412	Dott. F. Niceta, Palermo.	Zinsser & Co., Inc., Hastings-on-Hudson.	New York	June 14, 1905	Pure leaf.	7.54	7.62	.75	55.7	52.7	3.0	20.4	32.3	1.7	6.6	do.	Do.
413	do.	do.	do.	June 30, 1905	do.	8.00	7.72	.34	55.7	51.8	3.9	18.6	33.2	1.5	4.8	do.	Do.
414	Francisco Basso & Co., Palermo.	H. M. Rau, New York.	do.	June 29, 1905	Guaranteed pure leaf, extra ventilated.	7.44	6.89	.63	51.7	48.5	3.2	19.7	28.8	3.3	9.3	Some lentiscus.	Do.
415	Unknown.	do.	do.	May 24, 1905	Warranted pure.	7.21	8.06	1.75	53.2	48.7	3.5	20.1	29.6	2.7	8.8	Sumac only.	Do.
416	P. Savona & Co., Palermo.	A. Klipstein & Co., New York.	do.	June 6, 1905	Warranted pure leaf.	8.15	6.69	.66	54.5	50.4	4.1	19.2	31.2	1.7	5.5	Considerable stem tissue.	Do.

417	Unknown.....	H. M. Rau, New York.	May 31, 1905	Pure, first quality	7.73	7.44	1.06	55.2	51.9	3.3	20.1	31.8	1.7	5.2	do.....	Do.
432	R. Orto di Palermo, Palermo.	Bureau of Chemistry.	1905	<i>Cordia alliodora</i>	8.07	4.76	.26	45.9	41.7	4.2	22.6	19.1	14.5	23.7		
433	do.....	do.....	1905	<i>Tamarix africana</i>	10.56	10.59	1.19	43.5	39.7	4.8	23.8	15.9	31.6	39.6		

^a Received from J. S. Young, Baltimore, Md.

SUMMARY AND AVERAGES.

Samples.	Mole- ture.	Ash.	Sand.	Total ex- tract.	Sol- ble ex- tract.	Insol- uble ex- tract.	Non- tan- nins.	Avail- able tan- nins.	Color in one- half per cent solution.	
									Red.	Yel- low.
All samples:										
Average.....	7.79	8.18	1.41	52.5	48.4	4.1	19.5	28.8	3.1	10.7
Maximum.....	9.46	10.95	3.05	59.2	55.4	6.7	22.7	35.1	11.2	27.8
Minimum.....	6.34	6.12	.28	45.5	41.8	2.6	17.1	19.6	.8	4.7
Pure sumac:										
Average.....								31.9	2.1	9.4
Maximum.....								35.1	3.5	12.8
Minimum.....								27.4	.8	4.7
Adulterated samples:										
Average.....								26.6	4.1	11.8
Maximum.....								33.3	11.2	27.8
Minimum.....								19.6	1.9	5.6

INVESTIGATION OF 1907.

During 1905 and 1906 there was so much agitation of the question of sumac adulteration in the leather trade journals, and buyers had to become so well informed on the question, that it was thought advisable again to collect and examine samples. This was done in the spring of 1907, exactly as before, the samples being secured from all incoming consignments for a time before they passed into the hands of the consignee. The results of the examination of these samples are given in Table III, page 22.

DISCUSSION OF ANALYTICAL DATA.

Seventy-five per cent of the 53 samples examined were pure sumac or contained but traces of other material such as may have been present accidentally. The pure samples contained from 25.6 to 35.7 per cent and averaged 30.6 per cent of tannin, or 1.3 per cent lower than the 1905 samples. Microscopical examination showed that 25 per cent of the samples examined were adulterated with lentiscus, while one sample was pure lentiscus and another was Turkish sumac. The adulterated samples contained from 22.4 to 30.4 per cent and averaged 26.3 per cent of tannin or 0.3 per cent lower than the 1905 samples. These facts indicate that while adulteration is not so generally practiced as in 1905, individual shipments are apparently adulterated to about the same extent.

The lowest tannin content of all the samples was 22.4 per cent, the highest 35.7 per cent, and the average 29.4 per cent, the latter figure being practically identical with the average tannin content of the 1905 samples. Color tests were made on but few extracts, which indicated a somewhat higher average color in the pure 1907 samples than in the pure 1905 samples. Only 3 of the 13 samples which were found to be adulterated were labeled to that effect. The other samples were either labeled pure sumac or marked so as to give the impression that they were pure Sicilian sumac.

These results indicate that from 1905 to 1907 there was a decrease of about 16 per cent in the importation of adulterated shipments of sumac, but that those shipments which were sophisticated had been adulterated with practically the same percentage of lentiscus as formerly. The practice of labeling adulterated shipments "pure sumac" or "warranted 100 per cent pure sumac" appears to be as general as formerly, so that it is still absolutely necessary for a buyer who would be sure that he is purchasing a pure, high-grade sumac to have it examined.

COMMENTS BY IMPORTERS.

A copy of each analysis was sent to the consignee in all cases, but in some instances they were returned marked "Not found" and these firms could not be located. In a few cases the consignees made some comments, the most important of which are quoted below:

Your laboratory No. 198 ^a represents an importation of sumac with 25 per cent lentiscus, especially so ordered and imported for a New York firm with their knowledge that it was so admixed and was known in the trade as No. 2 goods. Laboratory No. 156, ^b showing an abundance of lentiscus * * * [is] the same as lot No. 198 * * * [and] has been imported with the condition of second quality, i. e., to contain 25 per cent lentiscus.

LEBER & SON.

* Leber & Son again stated on February 20, 1908, that the lot from which sample No. 1511^c was taken was imported mixed with lentiscus on the order of the purchaser.

Analysis of Nos. 124 to 128 * * * agree very closely with our own determinations. * * *

J. S. YOUNG & Co., LTD.

By CHARLES R. DELANEY, *Chemist*.

^a There were 280 bags in this invoice.

^b There were 140 bags in this invoice.

^c There were 70 bags in this invoice.

TABLE III.—*Chemical and microscopical examination of Sicilian sumac sampled in 1907.*

Laboratory No.	Consignor.	Consignee.	Collection of samples.		Statement on bags as to purity.	Chemical examination.						Microscopical examination.
			Place.	Date.		Total extract.	Soluble extract.	Insoluble extract.	Nonanims.	Available tannin.	Color in one-half per cent solution.	
						Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Red.	Yellow.
1509	Unknown	Fuorat Bros. & Co., New York	New York	Apr. 9, 1907	No statement	50.76	47.50	3.26	20.54	26.96		
1510	Unknown (Palermo)	Cove & Herbert, New York	do.	do.	Warranted pure sumac.	58.08	54.50	3.58	19.29	35.21		
1511	C. Wederkind & Co., Palermo	Leber & Son, New York	do.	do.	25 per cent lentis- cus.	49.33	45.66	3.67	20.21	25.45		
1512	Unknown	A. Klipstein & Co., Philadel- phia	do.	do.	100 per cent pure Sicily sumac.							
1513	C. Wederkind & Co., Palermo	Leber & Son, New York	do.	do.	Extra ventilated, non plus ultra.	49.90	46.77	3.13	20.59	26.19	3.0	11.0
1514	Mormino, Termini	H. M. Rau, New York	do.	do.	Extra pure	54.34	50.80	3.54	20.56	30.24	3.0	11.0
1515	C. Wederkind & Co., Palermo	Leber & Son, New York	do.	do.	Extra ventilated, steam ground.	51.94	48.80	3.14	20.80	27.90	2.6	10.4
1516	do.	do.	do.	do.	Extra ventilated, non plus ultra.	51.74	48.47	3.27	21.09	27.47	2.8	11.5
1517	do.	do.	do.	do.	Steam ground, non plus ultra.	49.51	46.13	3.38	20.53	25.60	2.7	10.0
1518	Unknown	do.	do.	do.	Guaranteed pure	52.59	48.64	3.95	20.71	27.93	3.0	14.0
1519	C. W. Saurmhaus, Palermo	A. Klipstein & Co., Boston	Boston	do.	100 per cent pure Sicily.	57.63	54.70	2.93	19.15	35.55	2.6	10.6
1520	Unknown	do.	New York		Pure Sicily, 100 per cent.	47.91	46.02	1.89	19.51	26.51	4.8	28.0
1521	do.	A. Klipstein & Co., New York	do.	Apr. 9, 1907	Per Sicily sumac, 25 per cent len- ticus							
1522	S. Falcone, Palermo	H. M. Rau, New York	do.	do.	Warranted 100 per cent pure.	55.47	51.96	3.51	20.17	31.79	2.0	9.8
1523	Unknown (Palermo)	O. S. Janney & Co., New York	do.	do.	Warranted pure.	49.66	45.36	4.29	19.77	28.59		
1524	P. Mancuso, Palermo	J. B. Moore & Co., Boston	Boston	Mar. 27, 1907	Warranted strict- ly pure.	58.42	54.51	3.91	19.97	34.54	2.3	12.0
1525	do.	W. L. Montgomery & Co., Boston.	do.	do.	Warranted pure sumac.	58.26	55.22	3.04	19.51	35.71	2.0	11.0

1226	Unknown	F. B. Cavell, New York.	New York.	do.	No statement.	54.79.46.94.4.85.22.7597.19	2.3.13.0	Do.
1227	do.	W. L. Montgomery & Co., Boston.	do.	do.	Guaranteed pure.	57.75.54.16.3.58.20.3183.85	1.9.9.2	Do.
1228	Mormino & Fi., Termini.	H. M. Rau, New York.	do.	Apr. 9, 1907	Ventilated, extra.	54.89.50.80.4.09.19.0431.76		Sumac with trace of lentiscus.
1230	Unknown.	F. R. Leonori & Co., New York.	do.	do.	Pure and prime 100 per cent.	46.92.43.98.2.94.21.8222.36		Composed largely of lentiscus, doubtful if any sumac present.
1275	Mormino & Fi., Termini.	H. M. Rau, New York.	do.	Apr. 18, 1907	Steam ventilated, extra pure.	54.30.82.34.1.96.20.2082.14		Do.
1276	G. Dalla & Fi., Palermo.	W. L. Montgomery & Co., Boston.	do.	do.	Guaranteed pure.	52.30.49.44.3.06.19.3230.12		Abundance of lentiscus.
1277	Unknown.	O. S. Janney & Co., New York.	do.	May 10, 1907	First quality ground, warranted pure.	47.97.44.09.3.88.19.9424.15		Sumac only.
1278	do.	do.	do.	do.	Guaranteed pure.	51.37.48.12.3.15.19.5828.54		Sumac with small amount of lentiscus.
1279	do.	A. D. Hitch & Co., New York.	do.	do.	Extra ventilated, steam ground, non plus ultra.	55.46.53.28.2.38.19.5633.72		Turkish sumac.
1281	Unknown.	Peck Bros., New York.	do.	do.	Pure Sicily sumac, 100 per cent.	50.52.46.98.3.94.19.3237.56		Sumac and abundance of lentiscus.
1282	do.	A. Kilpstein & Co., New York.	do.	do.	Pure Sicily sumac, 100 per cent.	52.5.50.2.2.4.16.3.30.9		Sumac only.
1283	do.	do.	do.	do.	Lentiscus to 20 per cent.	51.22.43.32.2.90.19.5828.74		Sumac with considerable amount of lentiscus.
1284	Salvatore Falcone, Palermo.	Marden, Orth & Hastings, Boston.	Boston.	do.	Pure masculino leaf, warranted 100 per cent.	52.32.46.14.3.18.18.7630.38		Sumac only.
1285	do.	Kilpstein & Co., Boston.	do.	Apr. 29, 1907	do.	52.40.49.18.3.22.20.3028.88		Sumac with considerable amount of lentiscus.
1286	Mormino & F., Palermo.	H. M. Rau, Boston.	do.	Apr. 23, 1907	Extra pure sumac.			Sumac only.
1287	P. Mancuso, Palermo.	W. L. Montgomery & Co., Boston.	do.	do.	Warranted pure.			Do.
1288	Unknown.	A. B. Hitch & Co., New York.	New York.	Apr. 24, 1907	Guaranteed pure.			Do.
2023	G. Dalla & Fi., Palermo.	W. L. Montgomery & Co., Boston.	Boston.	May 22, 1907	Guaranteed pure.			Do.
2024	Salvatore Falcone, Palermo.	Marden, Orth & Hastings, Boston.	do.	May 14, 1907	Warranted 100 per cent pure, best quality masculino.			Sumac with traces of lentiscus.
2025	Mormino & Fi., Termini.	H. M. Rau, New York.	New York.	May 13, 1907	Extra pure.			Sumac only.
2026	S. Falcone, Palermo.	W. L. Montgomery & Co., Boston.	Boston.	June 17, 1907	Warranted 100 per cent pure, best quality masculino.			Sumac and some lentiscus.
2027	C. Wederkind & Co., Palermo.	Leber & Son, New York.	New York.	do.	Extra ventilated, steam ground, non plus ultra.			Sumac.

TABLE III.—*Chemical and microscopical examination of Sicilian sumac sampled in 1907*—Continued.

Laboratory No.	Consignor.	Consignee.	Collection of samples.		Statement on bags as to purity.	Chemical examination.								Microscopical examination.	
			Place.	Date.		Total extract.		Soluble extract.		Insoluble extract.	Nontannins.		Available tannin.		Color in one-half per cent solution.
						Per cent.	Per cent.	Per cent.	Per cent.		Red.	Yellow.			
2038	Unknown.	F. R. Leonori & Co., New York.	New York.	June 17, 1907	100 per cent extra ventilated.										Lenticular, no sumac found.
2039	G. Dalla & Fi., Palermo.	W. L. Montgomery & Co., Boston.	Boston.	do.	Pure superior quality.										Sumac only.
2040	Unknown.	A. Klipstein & Co., New York.	New York.	do.	100 per cent pure Sicily sumac.										Do.
2041	do.	do.	do.	do.	do.										Do.
2042	do.	do.	do.	do.	Finest Sicily sumac, 20 per cent lenticular.										Sumac and lenticular.
2043	G. Dalla & Fi., Palermo.	W. L. Montgomery & Co., Boston.	Boston.	do.	Warranted pure sumac.										Sumac only.
2044	S. Falcione, Palermo.	A. Klipstein & Co., Boston.	do.	do.	Warranted 100 per cent pure, best quality maculino.										Sumac with abundance of lenticular.
2045	C. Wederkind & Co., Palermo.	Leber & Co., New York.	New York.	do.	Non plus ultra.										Sumac only.
2046	S. Falcione, Palermo.	H. M. Rau, New York.	do.	do.	No. 1, 100 per cent pure maculino leaf.										Do.
2047	Unknown.	Fuerst Bros., New York.	do.	do.	Finest quality warranted extra.										Sumac with trace of lenticular.
2048	S. Falcione, Palermo.	A. Klipstein & Co., New York.	do.	do.	100 per cent pure maculino leaf.										Sumac with some lenticular.
2049	Unknown.	O. S. Janney & Co., New York.	do.	June 6, 1907	First quality warranted pure.										Sumac with abundance of lenticular.
2050	do.	do.	do.	do.	do.										Do.

SUMMARY AND AVERAGES.

Samples.	Avall- able tannin.	Samples.	Avall- able tannin.	Samples.	Avall- able tannin.
All samples: Average..... Maximum..... Minimum.....	Per cent. 29.4 35.7 22.4	Pure sumac: Average..... Maximum..... Minimum.....	Per cent. 30.6 35.7 25.6	Adulterated samples: Average..... Maximum..... Minimum.....	Per cent. 28.3 30.4 22.4

DETECTION OF ADULTERATION.

NOTES ON THE MICROSCOPICAL EXAMINATION OF SICILIAN SUMAC AND ITS ADULTERANTS.

By B. J. HOWARD, *Chief, Microchemical Laboratory.*

The differentiation of pure and adulterated sumac by means of the microscope is not at all a new procedure, but it does not appear to be generally employed by the trade in this country. The work done in this laboratory indicates that this is a convenient and quick method of identifying certain of the common adulterants in Italian sumac leaves, and that in the detection of the most common adulterant, *Pistacia lentiscus*, no great experience is necessary to obtain reliable results. The examinations here reported include only commercial samples, most of which were in a powdered form, and hence no studies of sections were made. The investigations have been in progress since 1903. The paper by Priestman^a will be found very useful to beginners along this line, but the technique of the method as there described seems to leave something to be desired in the way of simplification. As will be shown, the technique adopted in this laboratory is quite different, and, it is believed, has some advantages over Priestman's.

APPARATUS AND REAGENTS.

The most important apparatus required is a good compound microscope giving a range of magnification of from about 75 to 200 diameters. Magnifications of 90 and 180 were actually used in the work here reported, but if approximately these powers are used, giving good definition, no trouble should be experienced. The instrument should have fine and coarse adjustments and a substage condenser with iris diaphragm. A mechanical stage with wide range of movement (about 2.5 cm or more) will be found very convenient, though it is not really necessary.

Microscope slides 25 by 75 mm (1 by 3 inches) and cover-glasses, round or square, are required, round covers of 0.75 inch diameter and from 0.17 to 0.25 mm in thickness, listed by some dealers in microscopical apparatus as No 2, are preferred. Some device for producing a small flame, such as a micro-bunsen burner or small alcohol

^a J. Soc. Chem. Ind., 1905, 24: 231.

lamp, is required. In addition to the above, a pair of teasing-needles, a pair of small forceps, and a scalpel should be secured.

As a clearing agent a chloral hydrate solution made up as follows was almost exclusively used: Chloral hydrate, 150 grams; water, 100 cc.

Among other reagents of occasional value the following should be noted: Alcohol of two strengths, 70 per cent and 95 per cent; two grades of glycerin, 100 per cent and 50 per cent (glycerin and water 1:1 by volume), and glycerin jelly are needed if permanent specimens are to be made, and this will almost always be done by careful workers.

The glycerin jelly is made up as follows: Best gelatin, 1.5 parts; water, 3 parts, and glycerin, 4 parts. Some persons^a prefer to use only 1 part of gelatin, since it gives a jelly more easily worked than the amount mentioned. Soak the gelatin in the water until it is soft, add the glycerin, and heat over a water bath, finally adding two or three drops of carbolic acid as a preservative.

TECHNIQUE.

The difficulty encountered on examining specimens mounted in water or glycerin direct is due to the fact that they are too opaque and contain considerable air. Some means of clearing the fragments are necessary. Priestman^b treated the sample with nitric acid, which attacked the more delicate tissues of the leaf first, and if the action was stopped at the right time, the leaf epidermis could be mounted as nearly clean tissues. This method is laborious if a large number of samples is to be tested, and seems to require considerable judgment as to just the stage at which the action is to be stopped, and hence is not desirable unless one is very familiar with microscopical technique.

In this work the chloral hydrate solution before mentioned was used. A small amount of the specimen is placed upon a slide with two or three drops of the solution and gently heated to boiling over the micro-bunsen burner or alcohol flame and kept gently boiling for about one minute. If the chloral hydrate solution boils away before the heating is finished, a few drops more are added, for if the specimens become dry the object of the treatment is defeated. After the boiling is completed the specimen is allowed to cool down somewhat, a cover-glass is placed over it and the specimen is ready for examination. If too much of the original specimen has been used, the mass will be too dense to give satisfactory results. A few tests, however, will demonstrate to the worker the most satisfactory amount

^a Clark's Practical Methods in Microscopy, 2d edition, 1896, p. 243.

^b Loc. cit.

to use. It is well to make several slides from the specimen, so as to get a good idea of its character.

Another method of procedure which some may prefer, and which lends itself readily to the examination of finely powdered samples, is the following: Place in a test tube a portion of the sample equal in size to a hazelnut, add a few cubic centimeters of the chloral hydrate solution and boil slowly for two or three minutes, allow to stand until the larger pieces have settled to the bottom and then remove a part of them with a pipette and mount on a slide in the usual manner.

Such treatment is all that is necessary in the preparation of samples for immediate examination. It is often desirable, however, to prepare specimens for future reference. For this purpose the specimen is cleared with chloral hydrate as described, the excess liquid is removed by a piece of filter paper, and then mounted in glycerin or glycerin jelly. To mount in glycerin, add to the moist fragments a small drop of the 50 per cent glycerin and after covering seal with a good microscopical cement. In order to mount in glycerin jelly, the sample is cleared and the excess chloral hydrate solution removed as previously directed, a tiny drop of 50 per cent glycerin is mixed with the moist fragments, and then a small piece of glycerin jelly (about a quarter of the size of a pea) is placed on the slide. The whole is gently heated until the jelly melts, and the fragments are mixed with the jelly by means of a teasing-needle or scalpel, care being exercised not to make bubbles in the mass, as they are difficult to remove. Care should also be taken not to heat the glycerin jelly on the slide enough to produce bubbles.

Permanent samples can be made, after clearing in chloral hydrate, by dehydrating in alcohol, clearing in xylol, and mounting in xylol canada balsam. This method is not satisfactory, however, unless the sample is stained; and with many small fragments, as is usual in a powdered sumac, this step is somewhat difficult and tedious.

SOME HISTOLOGICAL FEATURES.

A short description of the most characteristic histological features of sumac and the most important of its adulterants may be of value. Although written descriptions and photographs aid greatly, in beginning such investigations the microscopist should, of course, first work on samples of known purity, then on known mixtures, and finally on mixtures of a content unknown to him but prepared from authentic samples.

SICILIAN SUMAC (*Rhus coriaria*).

The upper epidermis of *Rhus coriaria* (Pl. I, fig.1) is made up of cells about 35μ in diameter (varying from 20μ to 50μ). They appear in the surface view to be bounded by walls with fairly straight sides—

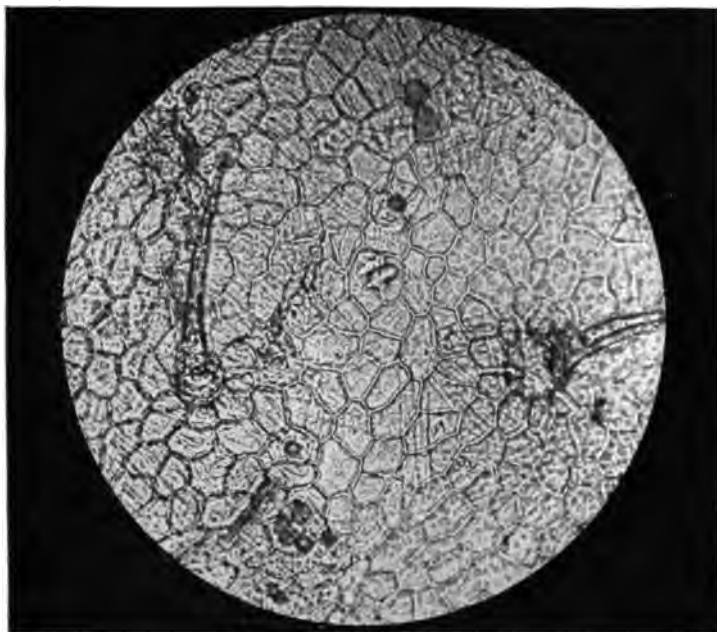


FIG. 1.—SICILIAN SUMAC (*RHUS CORIARIA*). UPPER SURFACE. $\times 150$.

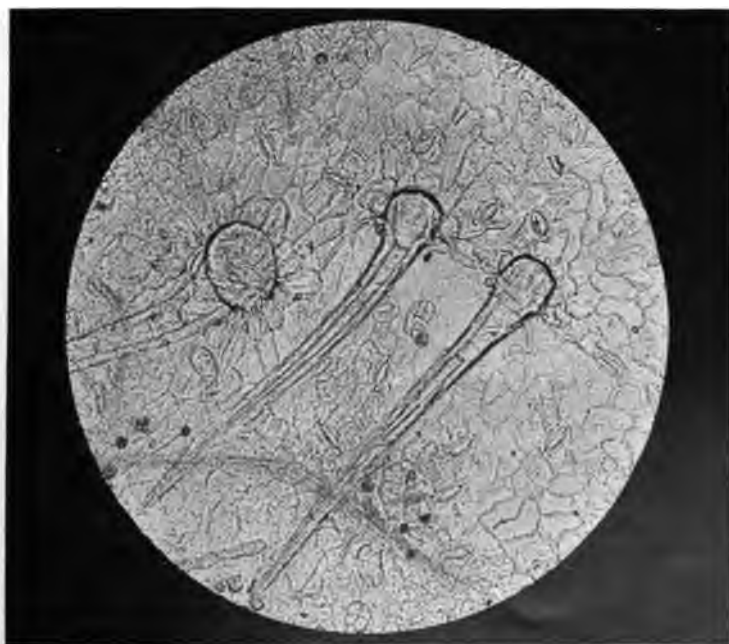


FIG. 2.—SICILIAN SUMAC (*RHUS CORIARIA*). LOWER SURFACE. $\times 150$.

that is, the individual segments of the periphery are but little distorted or curved. The walls are thin and have a slight beading due to deep, broad, regular pits, while the corners come down to quite sharp definite angles. There are present an abundance of horn-shaped hairs (trichomes) from 50μ to 400μ long and from 35μ to 70μ diameter at the base. Although the cavity in some of the hairs; especially the smaller ones, is simple, in many of them it is divided by transverse septa into two or three chambers. The epidermal cells adjoining each trichome are commonly from 8 to 14 in number, though these limits are at times exceeded. They are much smaller in average size than those of the intra-trichome regions. The cells of the under epidermis (Pl. I, fig. 2) are somewhat smaller than those of the upper epidermis, and the walls are much more bent or curved, giving the cells very irregular outlines. The beading of the walls is of about the same prominence as in the upper surface. On the under surface are two kinds of trichomes: (1) horn-like forms similar to those on the upper surface but usually longer; (2) glandular forms of from 3 to 4 cells, raised on a single-celled stalk, the whole forming a club-shaped structure. The horn-like trichomes of both the upper and lower sides of a leaf have commonly a slightly warty surface. The lower epidermis is also furnished with many stomata or breathing pores, but there is no such regularity in the number and arrangement of the adjacent epidermal cells as in the case of the lentiscus. Rosette crystals of calcium oxalate are often visible in the leaf tissue when viewed from either side, though some leaves show but few or none. An excess of stems is detected by the presence of fibrous tissue in greater amount than in good normal samples. Many fragments of the powdered sumac leaf will show only the trichome scars, since in grinding they are frequently broken off.

LENTISCUS (*Pistacia lentiscus*).

The method of clearing by chloral hydrate has a tendency to produce clearer tissue in *P. lentiscus* than with *R. coriaria*. Both surfaces of *P. lentiscus* are free from trichomes. The upper epidermis (Pl. II, fig. 1) is made up of cells having very conspicuous walls. The outlines of the walls are straight and at the angles, instead of coming down to sharp points, are slightly rounded, giving to them a very distinctive appearance and one not to be confused with the surface appearance of *R. coriaria* or any of its other common adulterants. This point is not so clearly shown in the photomicrograph as in the specimens themselves since this feature was subordinated to producing the best general effect, the latter being much more important in its identification. The cells vary in width from 17μ to 30μ .

The under epidermis cells (Pl. II, fig. 2) have not quite so prominent walls as those of the upper layer and the outline is more inclined to be wavy. The limits of variation in diameter also exceed those of the upper surface. From six to ten cells are radially grouped around each stoma. In the ordinary clearing process these cells, together with the stomata, commonly clear up more perfectly than the rest of the epidermal cells, thus giving to the specimen when viewed under the microscope with the objective slightly out of perfect focus a mottled appearance which is very characteristic.

TAMARISK (*Tamarix africana*).

This material cleared in chloral hydrate is more brownish in color than the species previously mentioned. The most characteristic feature observed is a papillæ-like appearance on the surface of the leaves distinguishing it from the other plants studied. This is best observed on fragments which lie partially on edge, in which position the little protuberances are readily seen. (Pl. III, fig. 1.)

SMOOTH SUMAC (*Rhus glabra*).

Though this species is not very commonly found if at all in *Rhus coriaria* as imported into this country, it is one of the possible adulterants that should be kept in mind. On neither surface of the leaf are the horn-like trichomes present. The cells on the upper epidermis resemble those on the upper epidermis of the *R. coriaria*, being ordinarily from 25μ to 52μ across. The beaded cell walls noted in the case of the *R. coriaria* are very pronounced in the *R. glabra* (Pl. III, fig. 2), and, together with the absence of trichomes, have been used as the basis of its identification.

The under surface of the leaf has epidermal cells and stomata and glandular hairs very much like the *R. coriaria*, but the cell walls do not generally show quite so much undulation. The fact that the horn-like trichomes are absent from both sides of the leaf is of additional service in the identification of whole or rather coarsely ground material, but in very finely ground samples can not be relied upon with certainty.

CHEMICAL DETERMINATION OF ADULTERANTS.

In the detection of adulterants it is customary to place dependence only on the microscopical examination of the sample, no chemical tests being regarded as of practical value for this purpose, although Proctor^a states that any sumac infusion rendered turbid by bromin water is open to grave suspicions. Work in this laboratory has shown that while pure sumacs are not as easily precipitated, requiring more bromin water than lentiscus extracts do, both the sumac

^a Principles of Leather Manufacture, 1903, p. 272.

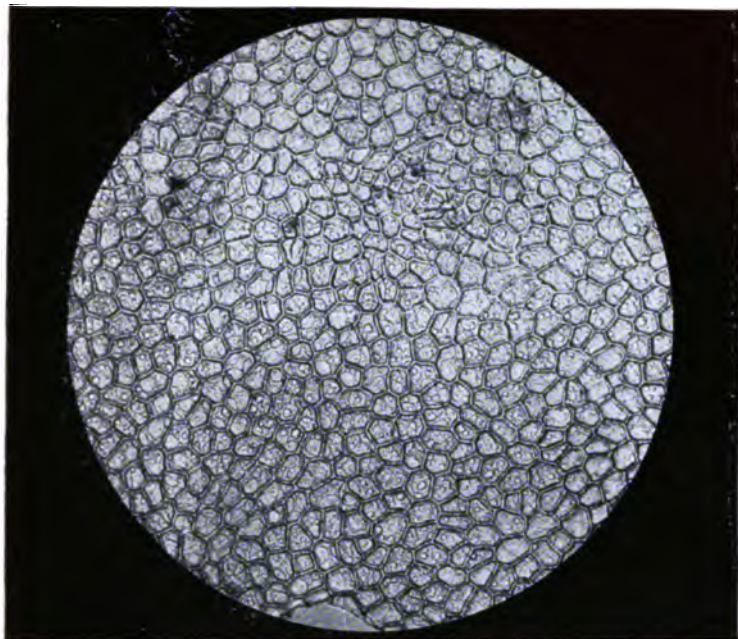


FIG. 1.—LENTISCUS (*PISTACIA LENTISCUS*). UPPER SURFACE. $\times 150$.

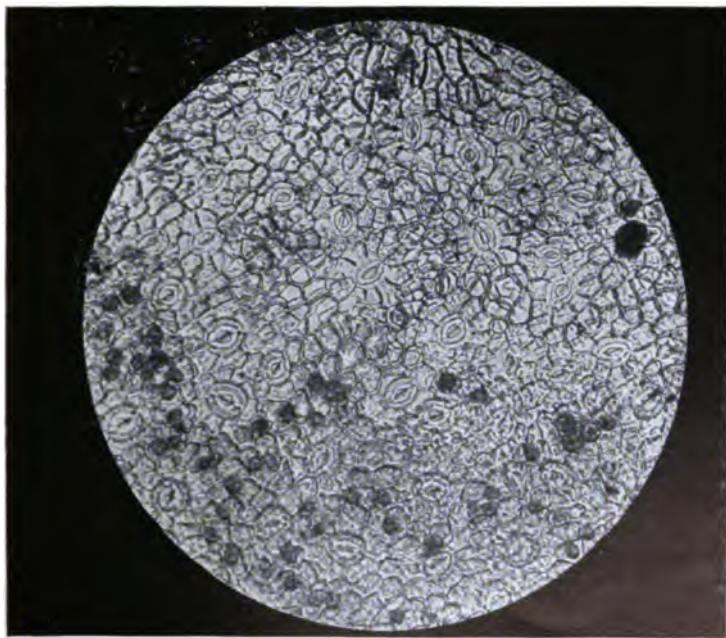


FIG. 2.—LENTISCUS (*PISTACIA LENTISCUS*). LOWER SURFACE. $\times 150$.

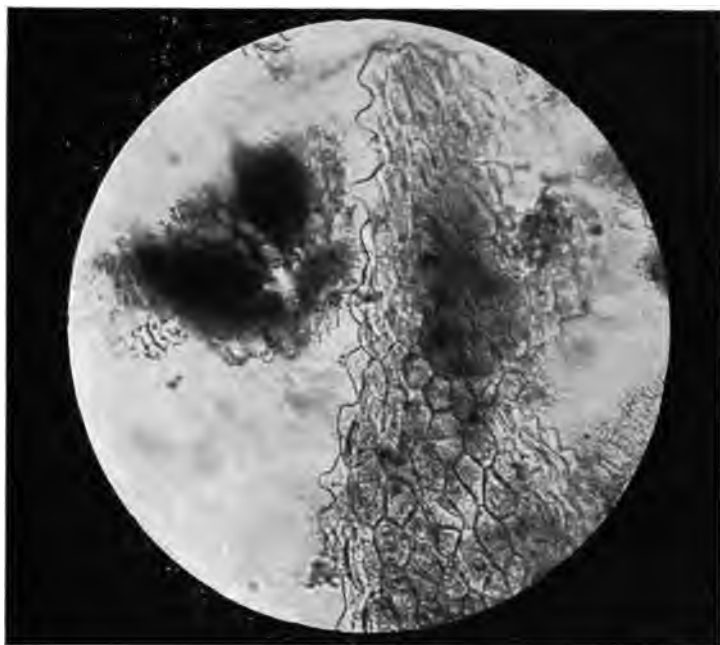


FIG. 1.—TAMARISK (*TAMARIX AFRICANA*), SHOWING PAPILLÆ OF EPIDERMIS. $\times 150$.

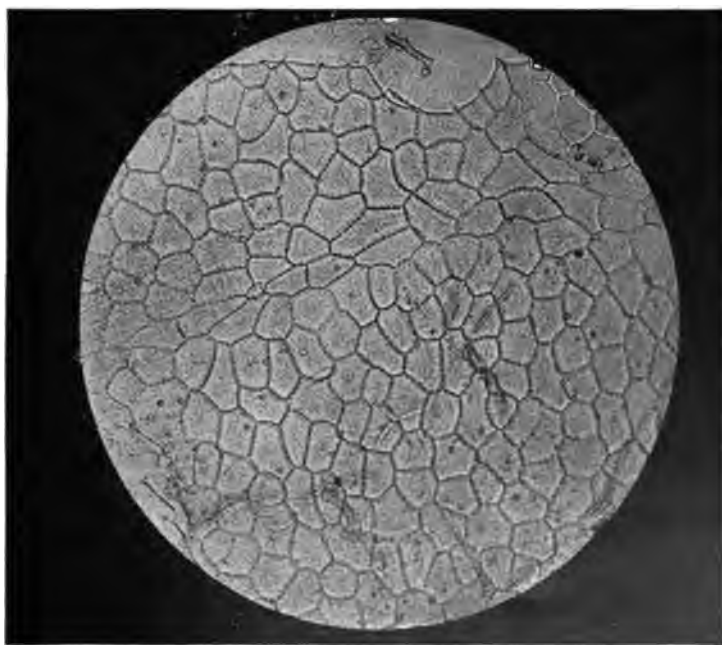


FIG. 2.—SMOOTH SUMAC (*RHUS GLABRA*). UPPER SURFACE. $\times 150$.

and its adulterants give a precipitate on treating with a quantity of saturated bromin water, and, as a consequence, but little reliance can be placed on this test. One of the most reliable indications of adulteration is the color of the dried sample. If *lentiscus* is present it will darken greatly on heating, becoming a dirty light brown with a tinge of red, while pure sumac only turns a slightly darker yellow. The experienced analyst, having a pure sumac for comparison, can pick out in nearly all cases samples adulterated with *lentiscus*. That this test agrees well with the microscopical examination is shown by the last column of Table II, where the purity of the samples as indicated by the color after drying is given. Of a total of 91 samples examined, 82 agreed with the microscopical test, 3 were doubtful, and 5 were erroneous. Moreover the color of the extract has been found a valuable indication, samples adulterated with *lentiscus* giving a dark reddish extract, easily distinguished from pure sumac. As a rule, therefore, the experienced analyst can distinguish by means of the color of the extract and the dried material those samples which are adulterated with *lentiscus*, but if there is any uncertainty a microscopical examination must be made.

Neither the percentage of ash nor of sand is an indication of adulteration with *lentiscus*, as this leaf does not differ materially from sumac in these particulars. The samples of leaf and ground sumac contained on an average 1.41 per cent of sand, the highest amount found being 3.05 per cent. Assuming that there was no sifting out of sand in transit, there was no evidence of willful addition of sand to these samples, although several indicated that they were but imperfectly winnowed or ventilated. There was less than 1 per cent of sand in the unground leaf, while 106 samples of ground leaf averaged 1.62 per cent and 15 contained more than 2 per cent. Therefore 2 per cent of sand may very properly be considered the maximum sand content of a well-ventilated ground sumac. A larger content of sand indicates that the samples have been carelessly prepared.

EXTENT OF ADULTERATION.

From 25 to 41 per cent of the invoices of Sicilian sumac imported into the country are adulterated, and this adulteration is effected almost exclusively with lentiscus. These adulterated shipments are, as a rule, so labeled as to convey the impression that they are pure Sicilian sumac. It is sometimes claimed that shipments of sumac are mixed with lentiscus in accordance with the order of the importer. In such cases the consignment should be properly labeled indicating the amount of lentiscus used. The tannin content is from 2 to 7 per cent lower in the adulterated samples than in the pure sumacs, averaging about 4.5 per cent lower, and the color of the extract prepared from them is much darker than that of pure sumac extracts. While to the experienced analyst the color of the extract or of the dried material is generally indicative of the purity of the sample, only microscopical examination can definitely determine this question.

The adulteration of Sicilian sumac is of more importance than is indicated merely by a lower tannin content, otherwise American sumac could be used at a much smaller cost. When high-grade, light-colored leathers or durable sumac-tanned leathers are required, as for instance in bookbinding, adulteration results in discoloration and destruction of the leather in a much shorter time than when pure sumac is employed in tanning, and the money loss thus occasioned is many times the difference in cost between a pure and an adulterated sumac.

Aside from any ethical consideration, there is absolutely no advantage to the tanner in the purchase of adulterated sumac because, as a matter of fact, the tannin in such sumac costs more for a given amount than when bought in pure sumac. Thus taking the current quotations of from \$71 to \$72 per ton for sumac containing 29 per cent of tannin, \$70 to \$71 for 28 per cent, and \$69 to \$70 for 27 per cent, the tannin costs from 12.2 to 12.4 cents, from 12.5 to 12.7 cents, and from 12.7 to 13 cents per pound, respectively. That is, the tanner is making a lower grade leather at a greater cost when using adulterated sumac. Finally, as there may be a variation of as much as 10 per cent in the tannin content of pure sumac, it should always be bought on the basis of its tannin content, and if adulterated should be so labeled.

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BUREAU OF CHEMISTRY—BULLETIN No. 118.

H. W. WILEY, Chief of Bureau.

UNFERMENTED APPLE JUICE.

BY

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1908.

LETTER OF TRANSMITTAL

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., July 7, 1908.

SIR: I present herewith a manuscript prepared by Mr. H. C. Gore on the preservation of fresh fruit juices, apple juice being used in these experiments. This is an industry of increasing importance in the United States, and it is only necessary to its still more rapid development that some unobjectionable method for the preservation of such juices be practiced. There is a continuing objection to the use of chemical preservatives in products of this kind, and the work here presented shows that these juices may be perfectly preserved, either in small or in large quantities and with a much better flavor, by means of sterilization alone. The work is of a practical character, having been conducted on a scale which can easily be extended to commercial proportions.

Mr. W. A. Taylor, pomologist in charge of field investigations, Bureau of Plant Industry, cooperated in the work by advising as to the selection of the varieties of apples employed, by serving on the committee of experts who judged of the quality of the completed product, and in other ways, giving valuable advice whenever the pomological features of the investigation were under consideration.

I recommend that this report be published as Bulletin No. 118 of the Bureau of Chemistry.

Respectfully,

H. W. WILEY,
Chief of Bureau.

Hon. JAMES WILSON,
Secretary of Agriculture.

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UNFERMENTED APPLE JUICE

INTRODUCTION.

The process of sterilizing, so extensively employed with other products, is not widely practiced with apple juice, probably because information is lacking as to methods of handling and the character of the product which may be obtained.

In an extensive treatise on cider making by a British investigator, F. J. Lloyd,^a sterilization is not mentioned as a practical process. Warcollier^b states that no method of sterilizing the fresh must has yet been commercially used in France. A report from the American consul at Munich gives a method of sterilizing employed there. It applies, however, only to bottled apple juice. In America methods have been developed for the preparation of bottled apple juice, but they have received no general application.

With a view to gaining information as to methods of sterilizing apple juice, experiments were conducted during the season of 1906^c in Nebraska and continued during 1907 at Washington, D. C. Apple juice is found to be a product which it is easy to sterilize, while conserving to a large extent the delicious flavor of freshly expressed juice. The process is inexpensive and capable of application on a small or large scale. If desired, the product may be carbonated before use, and when so treated closely resembles apple cider in which the natural fermentation is just beginning.

The term "sterilization" as used herein indicates that the fruit juices treated as described do not develop any bacterial or fungous growths within the period usually elapsing before their consumption. The temperatures employed, however, are those commonly used in the process termed "pasteurization."

The various phases of the investigation will be discussed, experiments having been made with wooden, tin, and glass containers, with different methods of clarifying and carbonating, and with the use of preservatives.

STERILIZATION OF APPLE JUICE.

IN WOOD.

These experiments were made to develop a method for sterilizing apple juice in wooden containers so that it might be marketed with-

^a Report on the Results of an Investigation into Cider Making, etc., 1893-1902. London, 1903.

^b Les méthodes scientifiques dans l'industrie du cidre. Rev. gén. sci. pures et appliquées, 1907, 18:778.

^c Yearbook of the U. S. Department of Agriculture, 1906, p. 239.

out the use of chemical preservatives, such as benzoate of soda, which is commonly used at present. Aside from hygienic reasons, experiments (see p. 19) have shown that the use of benzoate of soda is far from being a satisfactory means of preserving apple juice. The objections urged against sterilizing are (1) that a "cooked" taste is added to the juice, greatly injuring the flavor, and (2) that it is impracticable to hold the juice sterile for more than a limited period. These objections have been met. The investigations here reported demonstrate that only a slight cooked taste is produced by the heat treatment required and that it is a simple matter to protect the juice from inoculation after sterilizing.

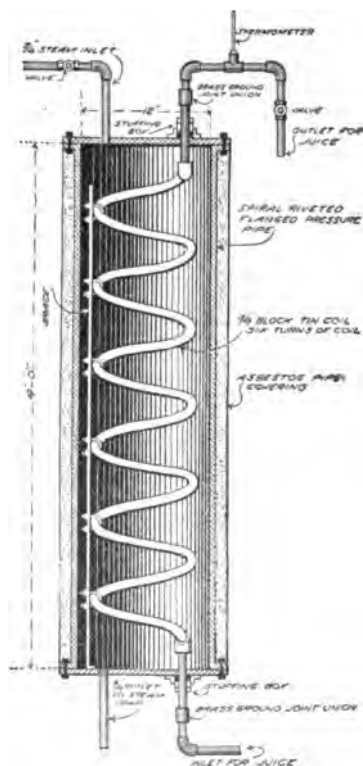


FIG. 1. - Pasteurizer for apple juice.

EXPERIMENTS WITH BARRELS.

In the experiments of 1906^a on sterilizing in barrels, it was found that they could be successfully used as containers when it was desired to keep the juice sweet for a few weeks.

In one experiment two 50-gallon barrels were thoroughly cleaned, well steamed, and filled with the juice heated to between 149° and 158° F. (65° and 70° C.). In sealing a cask which is full of hot liquid, air should be allowed to enter during cooling to destroy the vacuum caused by the contraction of the liquid. Unless this is done, a severe strain is put on the cask, greatly increasing the danger of contamination. In this experiment, instead of at first driving in bungs to close the barrels, clean cotton plugs were used. When the casks and contents were cool, the

plugs were removed and wooden bungs which had been sterilized by soaking in alcohol were quickly inserted. The juice kept for ten days without showing fermentation.

In this experiment the pasteurizer shown in fig. 1 was employed and proved to be a very useful machine, capable of heating the juice with perfect control of temperature at any desired rate up to several hundred gallons per hour. This pasteurizer was built for about \$50.

^a Loc. cit.

EXPERIMENTS WITH KEGS.

Further experiments on sterilizing in wood were made during the season of 1907-8, and the results show that wooden containers can be successfully employed for fairly long periods of time. In these experiments, 10-gallon kegs made of No. 1 white-oak stock, costing \$1.05



FIG. 2.—Small pasteurizer for sterilizing apple juice in kegs.

each, were used. A pasteurizer of smaller size was employed, similar to the one shown in fig. 1, but built of lighter material and costing about \$12 (fig. 2). The results were satisfactory and demonstrate that this model will be found useful in work conducted on a small scale.

The kegs were prepared for use as follows: They were paraffined on the outside by dipping in a bath of melted paraffin which was heated to about 120°C. (248°F.) by means of a steam coil. The arrangement is shown in fig. 3. Steam was run into the keg for about three minutes, when it was allowed to cool somewhat, and sulphured by lowering into each keg a small crucible filled with burning sulphur. The kegs were allowed to stand closed overnight, and in the morning just before filling they were steamed out for three minutes and well rinsed, removing practically all of the sulphur. It was found that a longer period of steaming melted the paraffin on the outside of the kegs.

The juice was heated by running through the pasteurizer at from 65° to 70°C. and was delivered directly into the kegs. As each was filled, it was closed in the following way: A wooden bung which had been paraffined and then dipped in alcohol was placed in the bunghole. The quarter-inch hole in the center of the bung was stuffed with cotton and the bung was driven into the keg (fig. 4a).

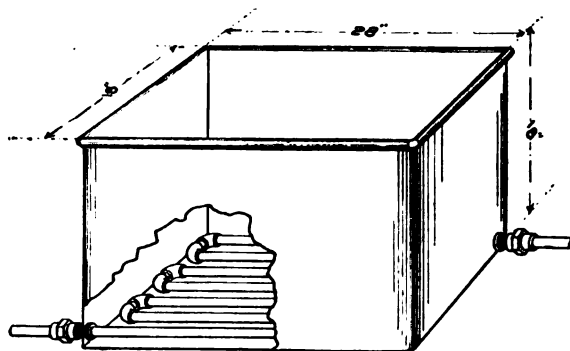


FIG. 3.—Apparatus for paraffining kegs.

was removed and another plug immediately inserted and saturated with alcohol. The cotton is stuffed into the bung before the bung is inserted in order to prevent the entrance of organisms while it is being driven in, and is replaced by a fresh plug of cotton afterwards, because

it usually becomes saturated with juice during the driving. This plug of cotton, sterilized by alcohol, prevents access of organisms during cooling, the air sucked in on account of the contraction of the juice on cooling being filtered through the cotton. When the juice had cooled the cotton plug was cut off at the surface of the bung, the portion of the plug remaining was wet again with alcohol, and a wooden skewer, fitting the hole in the bung closely, was sterilized by soaking in melted paraffin, then in alcohol, and driven into the hole, forcing the cotton plug out. In this way the cotton plug was replaced by a sterilized wooden plug without any chance for the entrance of organisms. The skewer was then sawed off even with the surface of the bung and smoothed over by a little melted paraffin (fig. 4, b and c).

Forty 10-gallon kegs were filled in this way, and of these 22 were kept unopened for more than six months, the juice in the remaining kegs being used for other purposes. The juice was prepared from

apples grown in the vicinity of Washington, being mostly of the Grimes (syn. *Grimes Golden*) variety, with some Winesaps, and while the fruit was sound and clean the juice was not of high quality, lacking in acidity and fruitiness. In no case, however, has the juice failed to keep well. The kegs were kept for the first two months in a warm cellar at about 20° C. Twenty-four were then subjected to a shipping test. Four lots of six kegs each were shipped by freight to Charlottesville, Va., and were held for periods of from two to four weeks in the cellar of W. B. Alwood, in charge of the enological investigations of the Bureau of Chemistry at that point, and then were returned to Washington. In no case was there any loss due to fermentation, although several kegs were injured in transit.

The first shipment left Washington on January 30, 1908, arriving in Charlottesville on February 3 in good condition. One keg of this lot was opened by W. B. Alwood and the following notes were made: "February 12, 1908. * * * I opened one keg of the first shipment of cider to-day and found it very good. Color yellowish straw, not translucent, but not muddy. Flavor excellent, barring slight cooked taste. Perfectly sweet and sound. Sp. gr. 1.053. I was able to take out with a siphon 35 quarts of fairly bright cider; 5 quarts were too muddy for use without sedimentation or filtering." On February 15 the remaining casks of this lot were returned to the Bureau of Chemistry at Washington, arriving in good condition. One of the kegs of the second shipment was leaking very slowly when received at Charlottesville, but the contents were found to be perfectly sound. The third shipment was received in good condition; but of the fourth shipment one keg was leaking, owing to a broken stave. Mr. Alwood calls attention to the fact that these kegs were too frail for the rough usage given freight, as the handlers drop them flat and thus spring the staves.

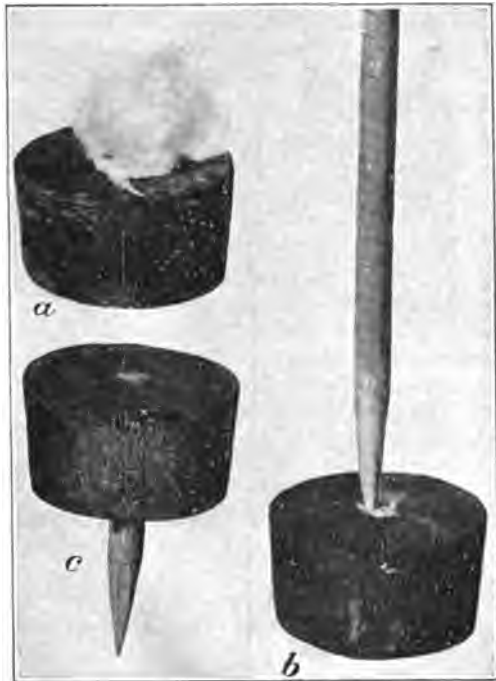


FIG. 4.—Wooden bung for cider barrels: a, Showing cotton plug. b, Skewer about to be driven in to displace cotton. c, Skewer in final position.

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All of the shipments from Charlottesville were received at Washington in good condition and have kept well subsequently.

ORGANOLEPTIC TESTS OF THE JUICE FROM KEGS.

On May 29 a keg was opened, the contents cooled to about 15° C. (59° F.), and a test made by a committee of three experts, H. W. Wiley, William A. Taylor, and J. A. Le Clerc, with a view to securing data as to the value of the product as a beverage for summer use. The opinions were as follows:

H. W. Wiley. Much more brilliant in color than Roxbury,^a though not perfectly bright. Somewhat unpleasant taste but not spoiled; do not like the flavor so well as that of the preceding sample. Slight flavor of barrel, but not unpleasantly strong. Sample is perfectly sound and sweet, but originally must have been inferior to that of the Roxbury.

After carbonating by running in a rapid stream of carbon dioxide (by means of which dilution of the juice was avoided) Doctor Wiley says: "Flavor much improved. It is now quite as palatable as Roxbury."

W. A. Taylor. I consider this a palatable and refreshing beverage. It has a perceptible cooked taste, but that is not objectionable at a temperature of 15° C. (59° F.). With one part of carbonated water to three parts of juice, the flavor is improved, as the cooked taste of the uncarbonated juice disappears. With one-half carbonated water and one-half apple juice, the fruit flavor fades away.

J. A. Le Clerc. A peculiar flavor, but can not detect any cooked taste. On the whole, it is a very good beverage. There is no alcohol, no perceptible acidity. I think the carbonating improves it and takes away the peculiar taste, the beverage still retaining enough of the apple flavor to make it acceptable; especially is this so with one part of carbonated water mixed with three parts of the cider.

It appears that, although the original juice used for this experiment was not of high quality, when sterilized, cooled, and carbonated it became a palatable and refreshing beverage.

IN CANS.

Experiments in the canning of apple juice during the season of 1906 consisted mainly in the determination of the proper conditions of processing, while during the past season the experiments have been chiefly devoted to investigations as to the quality of sterilized apple juice from different varieties of apples, and the maintenance of quality during storage in cans.

CONDITIONS OF PROCESSING.

A sterilizing process which was found in 1906 to be satisfactory consisted in heating the sealed cans in a water bath to 65° C. (149° F.). This requires from thirty to thirty-five minutes, the water in the bath being kept in constant agitation. The cans were then removed and allowed to cool. The flavor was but little affected by this treatment,

^a A sample of canned Roxbury apple juice was tasted by the committee on the same date (see page 13).

a very slight cooked taste being apparent. During the past season this treatment has not been found sufficient in all cases (see page 14), and the exact time of heating can not be considered as settled. The process just outlined should be made slightly more severe, either by increasing the temperature or by lengthening the time, or both. In no case, however, should the temperature exceed 70° C. (158° F.).

QUALITY OF JUICE OBTAINED FROM DIFFERENT VARIETIES OF APPLES.

The varieties employed in the season of 1907 were selected by William A. Taylor, pomologist in charge of field investigations, U. S. Department of Agriculture, with a view to securing as wide a range of quality as practicable. Varieties of known high quality were chosen as well as inferior apples; some were high in acid, yielding a rich juice, some contained only moderate amounts of acid, and one variety of sweet apples was employed. The fruit was purchased in lots of 6 barrels each, with the exception of one variety, the Kentucky Red crab, of which it was possible to secure only 3 barrels, and was held in common storage until fully ripe. They were then ground and pressed by a local cider maker, all decayed portions being removed before grinding. The fresh juice was first run through a separator to remove the bulk of the sediment, then canned, and sterilized by the method previously described.

When cooled, the juices were tasted by the committee of three experts before mentioned (page 10), who contrasted the sterilized product with the fresh apple juice. The varieties, locality where the apples were grown, and notes as to quality of juice after sterilizing are given in the following table, the notes being gathered from the opinions of the committee.

TABLE I.—*Organoleptic tests of sterilized juice from different varieties of apples.*

Variety.	Source.	Quality of sterilized juice.
Yellow Newtown (syn. <i>Albemarle Pippin</i>).	Waynesboro, Va.	Juice very palatable; distinguished from the fresh only by the slight cooked taste and a little bleaching or lightening of the color.
Ralls (syn. <i>Ralls Genet</i>).	Waynesboro, Va.	Very sweet; rather insipid; would probably blend well with some other juice richer in acid.
Ben Davis	Waynesboro, Va.	Quite unpalatable; lacking in distinctive apple flavor.
Winesap	Waynesboro, Va.	Very palatable; the fruity flavor somewhat impaired by sterilizing; slight bleaching noticeable; very little cooked taste.
Tolman (syn. <i>Tolman Sweet</i>).	Halls Corners, N. Y. ..	A very dark colored, thick juice; very slight cooked flavor and slight bleaching; very sweet and insipid.
Northern Spy	Halls Corners, N. Y. ..	Very fine in flavor; a fine rich juice, showing slight bleaching, and hardly detectable cooked flavor.
Baldwin	Halls Corners, N. Y. ..	High in quality, very palatable; slightly bleached and with slight cooked flavor.
Roxbury (syn. <i>Roxbury Russet</i>).	Halls Corners, N. Y. ..	A heavy, rich juice, very palatable; slightly bleached and with very slight cooked flavor.
Shockley	Waynesboro, Va.	Inferior in quality, but superior to Ben Davis; lacking in apple flavor and lacking in acid.
Gilpin	Waynesboro, Va.	Rather insipid, lacks character; a very palatable blend was produced by mixing with Roxbury.
Kentucky Red (syn. <i>Kentucky Crab</i> , possibly).	Mitchell, Ind.	Very light in color, almost water white; of delicious flavor, rather high in acid. Too light in color and probably too acid for general sale, but blends well with juices which are less rich in acid.

It is clearly shown that sterilized juices of high quality can be produced when first-class fresh juices are used. These tests show that the color is bleached somewhat, and a slight cooked taste is noticed after sterilizing. The general quality of the juice is, however, practically unaffected. The cooked or "boiled cider" taste in many cases was noticeable only when the unheated juice was tasted at the same time. To the writer the change in flavor resulting from sterilizing seems to be due to a slight but distinct loss in fruitiness, rather than to the formation of other flavors. The analyses of the uncooked juices are given in Table II.

TABLE II.—Composition of unfermented apple juice, 1907.

Variety.	Date of analysis.	Specific gravity.	Solids. ^a	Acid as malic.	Invert sugar.		Sucrose.
					Reducing sugar.	Total.	
Yellow Newtown (syn. <i>Albemarle Pippin</i>).....	1907. Dec. 17	1.0504	<i>Per cent.</i> 12.35	<i>Per cent.</i> 0.53	<i>Per cent.</i> 9.15	<i>Per cent.</i> 11.58	<i>Per cent.</i> 2.31
Ralls (syn. <i>Ralls Genet</i>).....	19	1.0564	12.82	.46	11.63	12.14	.48
Ben Davis.....	1908. Jan. 7	1.0492	12.05	.48	7.86	10.05	2.08
Winesap.....	10	1.0475	11.64	.46	9.06	10.02	.91
Tolman (syn. <i>Tolman Sweet</i>)..	10	1.0638	15.63	.13	9.92	13.95	3.83
Northern Spy.....	11	1.0608	14.90	.61	8.52	12.82	4.09
Baldwin.....	15	1.0584	14.31	.63	7.33	12.22	4.65
Roxbury (syn. <i>Roxbury Kusset</i>).....	16	1.0688	16.86	.70	7.46	13.81	6.03
Shockley.....	17	1.0457	11.20	.29	8.70	10.01	1.25
Gilpin.....	24	1.0547	13.41	.38	10.22	11.50	1.22
Kentucky Red.....	24	1.0608	14.90	.72	8.58	12.66	3.88

^a Calculated from the specific gravity, using the formula $S=245(s-1)$; see Browne, J. Amer. Chem. Soc., 1901, 23: 875.

The most palatable juices, Roxbury, Northern Spy, Kentucky Red, and Baldwin, contained about 12 per cent of total sugars, being quite rich in sucrose, and over 0.6 per cent of acid expressed as malic. A rich, rather acid juice thus appears to be most desirable for use in sterilizing. Besides the sugar and acid, the juice should possess a distinctive apple flavor, as was shown in the case of the Ben Davis juice, which, though quite rich, was far from being palatable.

ORGANOLEPTIC TESTS OF CANNED JUICE.

The canned processed juice was tested at intervals by the same group of experts whose opinions have been previously quoted. No deterioration in flavor was noted except in the cases in which the juices were not sterile (p. 14). Besides a slight bleaching effect observed in the juice from the plain cans as compared with juice held in "coated" cans, there was no difference detectable by the judges after nearly six months.

The palatability as a summer beverage of sterilized apple juice kept in tin containers was tested on May 29, 1908. Similar tests on

the juice from the kegs have already been reported (page 10). The comments on the canned juice of Roxbury, both uncarbonated and carbonated, were as follows:

H. W. Wiley.

Roxbury, uncarbonated: Somewhat turbid, sweet, and very palatable.

Roxbury, with addition of about one-fourth volume of carbonated water: Sweet and palatable but not so good as that carbonated before canning.

Roxbury, carbonated before canning: Somewhat turbid. Very agreeable and pleasant tasting; fine beverage, better than either of the other samples.

W. A. Taylor.

Roxbury, uncarbonated: A rich, heavy, satisfying juice with perceptible cooked taste and distinctive russet apple flavor.

Roxbury, with addition of about one-fourth volume of carbonated water: Distinctly better than the noncarbonated juice, being improved by the dilution as well as by the bite due to the carbonation. A very satisfying hot-weather beverage.

Roxbury, carbonated before canning: This is about like that carbonated by the addition of soda water, except that it needs dilution, being a little too rich and heavy.

Roxbury, carbonated before canning with the addition of carbonated water: Adding about one-fourth volume of carbonated water makes it practically like the uncarbonated sample when treated with soda water.

J. A. Le Clerc.

Roxbury, uncarbonated: Very sweet, and pleasant flavor. Free from alcohol and gas; pleasant aroma; no boiled-cider taste.

Roxbury, with the addition of carbonated water: When carbonated in the proportion of 1 part of carbonated water to 3 or 4 of cider, a marked improvement is observed. The drink is just sweet enough, and of a pleasant aroma, the gas of the carbonated water giving it the necessary life; no boiled taste.

Roxbury, carbonated before canning: Very similar to the uncarbonated, but it seems to have a little more life due to the carbonating.

Roxbury, carbonated before canning, with the addition of soda water: Very similar to the uncarbonated juice when treated with soda water under similar conditions.

From these observations it may be concluded that, although the fruit flavor is slightly diminished by sterilizing, the juice has evidently retained much of the characteristic apple aroma and flavor, and when carbonated resembles closely apple cider in the early stages of fermentation.

ACTION OF THE JUICE ON THE TIN OF THE CAN.

Apple juice quickly acts on the walls of the can, bringing out the crystalline structure of the tin. Tests showed, however, that but little tin is actually dissolved during the first few months after canning. The art of can making is now so far advanced that it is possible to exclude practically all metals except tin from contact with canned goods. Several kinds of cans are now on the market, at slightly advanced prices, whose covers are sealed on by the seaming process without the use of solder or acid, and the joint at the sides of the cans

is so made that no solder comes in contact with the interior. Some makers have gone a step farther and provide cans covered inside with a lacquer in order to protect the goods from contamination with tin, this being desired by the manufacturers not only for hygienic reasons but because tin has a deleterious effect on the color of many canned goods.

Two kinds of cans were used as containers for the several varieties of apple juice in this experiment. Both were of the type which practically excludes all metals except tin from contact with the juice. In the case of those designated in Table III as "plain" cans, the juice was exposed to the action of the tin; those designated as "coated" were covered on the inside with a lacquer. The amounts of tin found in various samples of juice and the time the sample had been in the can are given in Table III. These results indicate that while the lacquer was not a perfect protection it materially lessened the amount of tin dissolved, the decrease being nearly 50 per cent.

TABLE III.—*Tin found in canned apple juices.*

Varieties.	Date of analysis.	Time in can.	Kind of can.	Tin (SnO ₂) found.
	1907.	Days.		Gm. per 100 cc.
Yellow Newtown (syn. <i>Albemarle Pippin</i>).....	June 13	179	{ Plain.....	0.0080
			{ Coated.....	.0052
Northern Spy.....	do	154	{ Plain.....	.0058
			{ Coated.....	.0026
Roxbury.....	June 20	156	{ Plain.....	.0061
			{ Coated.....	.0033
Kentucky Red.....	do	148	{ Coated.....	.0059
Mixed varieties.....	do	627	{ Plain.....	.0156

SPOILAGE.

It has been stated (page 11) that the conditions of processing were not absolutely established, since some varieties spoiled in the cans, whereas others, sterilized under the same conditions, remained perfectly sound. The facts observed are as follows: The cans of Tolman some weeks after processing began to swell, and on opening there was a considerable evolution of gas. A butyric acid odor was present, and the juice had become ropy. The cans of the Shockley variety began to swell, some weeks after canning, but on opening showed no ropiness or bad flavor. The Ralls became ropy very slowly, but the flavor did not change materially, and there was no evolution of gas. The Ben Davis juice also became ropy, but no further evidence of fermentation was apparent, and the Gilpin juice showed the same phenomenon. The canning was done in a room which was used at times by others for experiments with dairy products, and it is quite possible that the juice was exposed to contamination not usual in ordinary work. Still, the results would indicate

that sterilization should be effected either by bringing the juice to a higher temperature than 65° C. or by heating for a longer time than thirty-five minutes. It will be noted that the three juices which are low in acid spoiled, namely, the Tolman, the Shockley, and the Gilpin, as well as those containing moderate amounts, as, for example, the Ben Davis and Ralls, plainly indicating the influence of acid in assisting sterilization.

IN GLASS.

In 1906 an extended series of experiments was conducted to determine the best treatment for sterilizing in bottles. The results showed that heating for one hour at 149° F. (65° C.) in a water bath gave good results, and that heating one-half hour at 158° F. (70° C.) was also a satisfactory process, allowing in each case a half hour for the contents of the bottles to attain the bath temperature. The products could be processed for as long as one hour at 158° F. without any marked deterioration, allowing a half hour for preliminary heating, thus making the total time in the water bath one and one-half hours. A very important consideration in the case of bottled apple juice is the removal of the sediment. The milk separator (see page 16) will remove the greater part of the sediment when operating on freshly expressed juice. It does not, however, remove all, so that a brilliant juice is not obtained when clarified in this way. The product is still slightly turbid and gradually deposits a sediment which much impairs the appearance of the cider. Further experiments on sterilizing in glass have been deferred, chiefly because it is felt that there is less need of investigation in this direction than along the line of preparing sterilized apple juice in wood and tin containers.

CLARIFICATION TESTS.

METHODS EMPLOYED.

Freshly expressed apple juice normally contains considerable quantities of insoluble matter. This is true of all apple juices which have been used except Kentucky Red, which was almost free from such material. This insoluble material consists largely of albuminous matter, starch grains, and yeast cells, together with some dirt particles, all of which settles on the bottom of the container, forming a thick, brownish layer. As has been stated, the simplest way of removing the greater part of this material is by the use of the milk separator, as was demonstrated in the experimental work of 1906. This method, however, can only be successfully practiced with absolutely fresh juice.

In the experiments of 1907, owing to the local conditions at the mill where the apples were pressed, it was necessary to grind at night,

and press on the following morning. The experiments on the clarification of the juice were not entirely successful, however, as the albuminous matter was so finely divided by the incipient fermentation that only partial clarification was possible. Large quantities of sediment were, nevertheless, removed by passing the juice through the separator, but the clarification was not so complete as that secured in 1906 when perfectly fresh juice was used. A handpower cream separator of the disk type was employed at that time, which collected the suspended matter in the juice in the bowl of the separator, while the clean juice ran out through the milk and cream screws. After being run through the machine, the heavier particles such as starch grains, or dirt particles, together with some of the albuminous matter, were found tightly packed in the lower part of the tubular shaft in the bowl of the machine, while a heavy layer of albuminous material collected on the inner side of the bowl and a lighter layer on the inner side of the bowl cover. The disks remained free from sediment. When the space between the disks and the sides of the bowl is quite filled with sediment, the flow from the milk screw ceases, and the machine should be cleaned. The juice from the milk screw is invariably considerably clearer than that from the cream screw. The reason for this is not apparent; the fact, however, was always observed. The juice from the cream screw is in turn much clearer than the untreated juice.

RESULTS OBTAINED.

An extended series of tests in 1906 established the following facts with regard to the method of clarifying by passing through a separator, using unfermented juice, and a machine of the size indicated:^a

First. The amount which may be run through the machine before it is necessary to stop and clean the bowl is from 25 to 40 gallons, depending on the quantity of sediment present in the juice.

Second. The rate at which the juice passes through the machine is about 45 gallons per hour, when a delivery tube of 450 pounds per hour (for milk) is employed. On fitting the separator with a delivery tube of 750 pounds' capacity per hour, less perfect clarification was effected than when the smaller delivery tube was used.

Third. But very little increase in the degree of clarification was secured when juice heated to from 140° to 158° F. (60° to 70° C.) was run through.

Fourth. When heated juice was allowed to stand overnight to cool and settle before passing through the separator, the supernatant juice contained much less sediment than the original juice and two to three times as much could be passed through the machine before cleaning became necessary as when unsedimented juice was used.

Fifth. Two separations are necessary when working with a separator of the size employed. The first treatment removes the bulk of the sediment, and the second takes out nearly all of the remainder.

Sixth. Running the juice more than twice through the separator improves the character of the product but little, as only very small amounts of the remaining suspended matter are removed.

^a Yearbook of the U. S. Department of Agriculture, 1906, p. 241.

Seventh. The best conditions, as worked out by experiment, for clarifying apple juice are as follows, working with a hand machine with a capacity for milk of 450 pounds per hour.

(a) The juice must be freshly expressed and, to be of high quality, should be prepared from sound, well-ripened fall or winter apples of suitable varieties.

(b) It should be received in a clean barrel or cask, which must not contain any fermentation residues. This point is very important, as experience has shown that the very fine deposit formed in fermenting juice can not be successfully removed by the separator, and this deposit is difficult to clean from the sides and bottoms of fermentation casks.^a

(c) In passing the juice through the separator, the necessary precautions as to oiling and starting the machine must be used, and the crank should be run at the rate of 45 turns per minute. Twenty-five to forty gallons of fresh juice can be run through before the capacity of the bowl for sediment is reached. The juice which comes through the milk screw should be collected separately.

(d) As soon as the milk screw becomes clogged the machine should be stopped and the bowl cleaned.

(e) The juice collected from the milk screw should be passed through again and that coming from the milk screw collected as before.

The clarification of 25 gallons of juice (using one machine of the capacity indicated and a juice containing sediment in such quantity that the run would fill the space between the disks and the sides of the bowl with sediment) requires about one hour and a quarter, the juice passing through the bowl twice.

These details are given in order to assist others who may wish to clarify in this way. Clarification by means of centrifugal force could probably be greatly improved if a machine designed primarily for the purpose were used.

CARBONATING.

Experiments in carbonating apple juice were carried on during 1906 to determine whether or not the palatability of the sterilized juice could be increased and the slight cooked taste be disguised.

METHODS EMPLOYED.

The following methods were tried in the work of 1906:

The juice was carbonated under slight pressure and then heated in bottles or cans. In the simple experiments conducted in connection with this work, the carbon dioxide (carbonic-acid gas) was secured from a firm dealing in soda-water supplies. It was obtained in liquid form in a steel cylinder furnished with a reduction valve and a gauge and delivery tube, so as to deliver at a pressure up to 30 pounds.

After clarifying, the juice was carbonated by pouring about 12 gallons of it into a clean keg and running in the gas up to a pressure of 15 pounds. The keg was provided with a thick pine bung, through the middle of which was bored a half-inch hole, which received the rubber delivery tube from the cylinder of compressed gas. The bung was soaked in water for a few minutes before use, so that it could be driven in to make a tight joint, and was so fitted that it projected beyond the surface of the keg and could be readily loosened when carbonation was finished. Carbon dioxide was ad-

^a This point is confirmed in the experiments of 1907 (see p. 15).

mitted before driving the bung in air-tight in order to expel the air which fills the space in the keg not occupied by the juice. The bung was then driven in by tapping with a hammer and more gas admitted. The keg was vigorously rocked so as to thoroughly agitate the juice and thereby accelerate the absorption of the gas.

The gauge was watched, the pressure not being allowed to go beyond 15 pounds per square inch. The juice used in the carbonating work was quite cool, ranging from 48° to 68° F. (9° to 20° C.) in the different experiments. From fifteen minutes to one-half hour was required to carbonate 12 gallons of juice. The stream of gas was then stopped, the bung cautiously loosened, the contents of the keg poured out, and the juice bottled or canned.

The gas remains for some time in the juice when under atmospheric pressure and only gradually diminishes in quantity, so that great haste in sealing the containers is not necessary. If the carbonated juice is to be sterilized in cans, they must be heated in stout frames to prevent the distortion of the can while hot and consequent bursting. The finished canned product bulges the ends of the cans to some extent, but not enough to cause permanent bending. The juice must not be too highly charged with the gas nor removed from the frames while still hot, or such bending, with consequent weakening of the soldered joints and bursting of the can, may occur.

Several varieties of apple juice were carbonated and then canned by this method during the past season, and it was found that the presence of the gas added an agreeable sparkle to the juice, at the same time, however, introducing a flavor foreign to fresh unfermented apple juice. If sterilized apple juice were sold at a soda fountain, it would be simple to add carbonated water, or cool the juice and run in carbonic-acid gas under pressure. In this connection it should be mentioned that apple juice acts rapidly on metals, particularly on galvanized iron, and if the juice is carbonated in tanks care should be taken that they are lined inside with tin rather than with any other common metal and that the juice is not kept in metallic containers.

USE OF CARBON DIOXID TO PREVENT MOLD.

It has been found that when sterilized apple juice is exposed to the air the organisms which usually develop are not those which produce the alcoholic fermentation but are molds which grow on the surface of the juice, giving rise to disagreeable flavors and soon making the juice undrinkable. Since the molds are usually aerobic organisms, it was thought possible to retard their development in most cases by maintaining an atmosphere of carbonic-acid gas over the juice. The following experiment was tried:

In June, 1908, about a gallon of sterile Northern Spy apple juice was placed in two large bottles, each bottle being about half full. The juice was poured from one bottle into the other, taking no precautions to protect it from contact with the organisms of the atmosphere.

One bottle was then stoppered and into the juice in the other bottle a rapid current of carbon dioxid was passed for about ten minutes, in this way partly saturating the juice with carbon dioxid

and displacing the air above the juice by the gas. This bottle was also tightly stoppered and the two were kept side by side in the laboratory. The following observations were made:

After three days a slight growth was observed on the surface of the juice which was untreated with carbon dioxid; no growth was apparent on the juice treated with gas.

On the following day thirteen colonies, several of which showed greenish groups of spores, were found on the untreated apple juice; none was found on the surface of the other. On June 8, three days later, there was a much greater development of spores and mycelia on the surface of the untreated apple juice, which had a flavor suggesting rotten apples. On the surface of the other juice there were found several filmy growths, but no development of organisms giving a disagreeable flavor. On removing the stopper there was a slight gas evolution and the liquid evolved carbon dioxid slowly, indicating that alcoholic fermentation was starting. No unpleasant flavor was apparent. A repetition of the experiment gave similar results, namely, the suppression of mold growths. It would probably be possible, in case the product is sold at soda fountains from small kegs, to saturate the juice and fill the air space above it with carbonic-acid gas, thus suppressing the development of the mold for a limited time.

Another method of eliminating mold on the surface of the liquid, or preventing fermentation, would be to open the package under sterile conditions and insert a sterile faucet such as has been described by Dodson^a of the Louisiana experiment station. This spigot is so arranged as to allow the entrance of air during the outflow of the liquid, the incoming air being filtered through cotton. If such a faucet is used for apple juice, however, it must be made of porcelain or wood.

EFFECT OF BENZOATE OF SODA AS A PRESERVATIVE.

Apple juice at present is largely sold in bulk, using a small amount of benzoate of soda to retard fermentation, one-tenth of 1 per cent being tolerated by regulation in the United States. An experiment was carried on to determine the effect of sodium benzoate when added in varying amounts to apple juice, with a view to finding out how long such juice would keep and what quality of juice resulted after standing for different lengths of time. For this purpose five 10-gallon kegs which contained sterile apple juice were opened and a culture of a pure yeast, supplied by W. B. Alwood, was added to each keg. Varying amounts of benzoate of soda were added and the kegs were kept nearly full and closed by tightly fitting cotton plugs.

^a Louisiana Agr. Exper. Stat., 1903, second series, Bul. No. 75, p. 256.

The cotton became wet with juice, however, and soured; the juice in the kegs in this way became infected with acid-forming organisms. Analyses were then made at intervals with the results shown in Table IV.

TABLE IV.—*Changes in composition during storage of apple juice containing various amounts of benzoate of soda.*

Description of sample.	Date of analysis.	Time of storage.	Specific gravity.	Solids.	Total acid as acetic.	Volatile acid as acetic.
	1907. Nov. 16	Days.	1.0499	Per cent. 12.22	Per cent. a 0.35	Per cent.
Original unfermented juice.....						
	1908.					
Fermented juice, no benzoate of soda....	Jan. 28	73	.9974	1.41	.71	0.49
	Mar. 18	123	1.0005	1.30	1.79	1.61
	May 26	192	1.0060	1.41	3.77	
Fermented juice +0.03 per cent of benzoate of soda.....	Jan. 28	73	1.0060	3.24	.70	.38
	Mar. 18	123	1.0076	3.03	1.53	1.47
	May 26	192	1.0103	2.56	2.99	
Fermented juice +0.06 per cent of benzoate of soda.....	Jan. 28	73	1.0184	5.66	1.15	.53
	Mar. 18	123	1.0135	3.94	2.08	1.81
	May 26	192	1.0215	3.91	4.84	
Fermented juice +0.10 per cent of benzoate of soda.....	Jan. 28	73	1.0502	12.48	.90	.39
	Mar. 18	123	1.0452	11.08	1.26	.71
	May 26	192	1.0176	4.73	2.59	
Fermented juice +0.15 per cent of benzoate of soda.....	Jan. 28	73	1.0517	12.44	.76	.25
	Mar. 18	123	1.0251	6.94	1.06	.53
	May 26	192	1.0213	5.16	2.77	

Description of sample.	Date of analysis.	Fixed acid as malic.	Alcohol.	Invert sugar.		Sucrose.
	1907. Nov. 16	Per cent.	Per cent.	Per cent. 9.29	Per cent. 10.21	Per cent. 0.87
Original unfermented juice.....						
	1908.					
Fermented juice, no benzoate of soda....	Jan. 28	b 0.25	5.39	.13	.24	.10
	Mar. 18	c .11	4.17	.11		
	May 26		2.03	.21		
Fermented juice +0.03 per cent of benzoate of soda.....	Jan. 28	b .36	4.55	1.59	1.73	.13
	Mar. 18	c .13	3.75	1.43		
	May 26		2.40	1.10		
Fermented juice +0.06 per cent of benzoate of soda.....	Jan. 28	b .60	3.12	3.61	3.73	.11
	Mar. 18	c .27	2.77	2.04		
	May 26		.35	2.02		
Fermented juice +0.10 per cent of benzoate of soda.....	Jan. 28	b .56	.42	9.64	10.13	.47
	Mar. 18	c .48	.76	8.52		
	May 26		2.53	2.40		
Fermented juice +0.15 per cent of benzoate of soda.....	Jan. 28	b .57	.09	9.80	10.16	.34
	Mar. 18	c .52	2.56	4.76		
	May 26		1.70	2.70		

a Calculated as malic acid. b Difference between total and volatile acids. c Determined analytically.

It will be noted that the juice which contained no benzoate of soda became practically dry during the first interval of 73 days, containing 1.41 per cent of solids and 0.24 per cent of total sugars as invert. The specific gravity fell to slightly less than 1. There was a formation of 5.39 per cent of alcohol and also formation of acetic acid, while the fixed acid suffered a slight loss. From this time on the usual aceti-

fication ensued, which was not complete on May 26, the time of the last analysis.

The juice which contained 0.03 per cent of benzoate of soda fermented considerably during the first interval, yielding 4.55 per cent of alcohol, a little acetic acid being formed at the same time. The juice, however, was far from being dry, 3.24 per cent of solids, including 1.73 per cent of total sugar, calculated as invert, being present. During the next interval there was a slight gain in total acid calculated as acetic, and a slight loss in alcohol. The sugar also diminished, and after one hundred and ninety-two days there were present 2.56 per cent of solids, 2.99 per cent of acid calculated as acetic, 2.40 per cent of alcohol, and 1.10 per cent of reducing sugar. The addition of 0.03 per cent of benzoate of soda, therefore, retarded the alcoholic fermentation, but permitted the alcohol formed to acetify.

Juice preserved by the addition of 0.06 per cent of benzoate of soda showed a greater retardation of fermentation, containing at all times considerably more solid matter, more sugar, and less alcohol. At the time of the last examination, however, it showed not only much less alcohol, but much more acetic acid than any of the other samples, still containing 3.91 per cent of solids, of which 2.02 per cent were sugars.

The juices containing 0.1 per cent and 0.15 per cent of benzoate of soda, respectively, showed during the first interval of seventy-three days practically no alcoholic fermentation, but increased considerably in content of total acid calculated as acetic. After one hundred and twenty-three days the juice to which 0.1 per cent of benzoate of soda had been added contained 11.08 per cent of solids, 1.26 per cent of acid as acetic, 8.52 per cent of sugar, and 0.76 per cent of alcohol. After one hundred and ninety-two days it had lost a large amount of its solid matter, but only small amounts of acetic acid and alcohol were present.

It was expected that the juice which contained 0.15 per cent of benzoate of soda would show less loss in solids than any of the other juices. This, however, was not the case. The reason for this may be that different organisms developed in the different barrels, and these were affected differently by the presence of the preservative.

Therefore, while benzoate of soda when added in quantities of 0.03 per cent and over retards alcoholic fermentation, eventually both alcoholic and acetic fermentation do develop, and an attempt to preserve apple juice as a beverage for any length of time by the use of benzoate of soda in quantities tolerated by the regulations would result in failure due to the development of acetic fermentation and consequent depreciation in flavor.

The alcoholic fermentation which would naturally take place in the absence of both preservatives and sterilization tends to exclude other

organisms, such as the molds and acetic acid ferments which require the presence of air for their development. The carbon dioxide produced by the action of the yeast on the sugar displaces the air above the juice in the container, and the air originally present in the juice is probably utilized by the yeast in its development so that such juice, if properly handled, yields a sound, fermented apple juice or cider. In the presence of preservatives, however, the yeasts are not permitted to develop and exert their effect of excluding other organisms, some of which will develop and produce bad flavors, rendering the juice unpalatable if not totally unfit for consumption. Sterilization, on the other hand, excludes the yeasts and the other organisms as well, so that the original flavor of the product, aside from a slight change due to heating, is maintained.

SUMMARY.

(1) The experiments described show conclusively that it is possible to sterilize apple juice in wooden containers, the product remaining sound for at least six months under actual observation. The precautions which must be taken to insure this are as follows: First paraffin the containers on the outside, then sterilize, and fill with juices heated to between 149° and 158° F. (65° to 70° C.); seal, taking measures to relieve the vacuum produced by the contraction of the juice on cooling by filtering the air through cotton. Twenty-four 10-gallon kegs successfully stood a severe shipping test, showing no loss due to fermentation of the juice. The juice so prepared was found to be palatable, and acceptable as a summer drink.

(2) It is demonstrated that apple juice can be successfully sterilized in tin containers, using the type of tin can sealed by the mechanical process, excluding all metals from contact with the juice except the tin of the can. Where lacquered cans are used the contamination with tin was reduced about one-half. Apple juices were canned and sterilized by heating in a hot water bath, up to the temperature of 149° F. (65° C.) for a half hour, and then were allowed to cool. These juices possessed only a slight cooked taste due to the heating and retained much of their distinctive apple flavor. It was found that from finely flavored apple juice a first-class sterile product could be made, while a poorly flavored apple juice yielded an inferior product. The process conditions mentioned were not quite thorough enough to sterilize all of the varieties canned. A slight increase in the temperature or time of processing, or both, should be made, the temperature not to exceed 70° C. (158° F.) in any case.

(3) The best treatment for sterilizing in glass was found to consist in heating for one hour at 149° F. or for one-half hour at 158° F. Heating for one hour at 158° did not produce marked deterioration in flavor,

a half hour being allowed in all cases for the juice to obtain the temperature of the water bath.

(4) It was shown that the great bulk of the insoluble material naturally contained in apple juice can be removed by means of a milk separator.

(5) It is possible to carbonate the juice slightly before canning or bottling, thus adding a sparkle to the product. A flavor foreign to fresh apple juice is also added, however, and uncarbonated sterile juice will resemble fresh apple juice more closely. Carbonating by the addition of water charged with carbon dioxid was considered by some to injure the flavor, lessening the characteristic fruit flavor by dilution. In the opinion of others a heavy, rich juice was improved both by the charge of carbon dioxid and by the consequent dilution. Experiments indicated that the danger of contamination by mold growths was lessened by maintaining an atmosphere of carbon dioxid above the surface of the juice after opening.

(6) It is demonstrated that benzoate of soda in quantities varying from 0.03 to 0.15 per cent (0.1 per cent being the maximum temporarily permitted by the food regulations), while it checks the alcoholic fermentation, allows other organisms to develop (notably the acetic acid ferment), whereby the palatability of the product as a beverage is destroyed.

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BUREAU OF CHEMISTRY—BULLETIN No. 119.

H. W. WILEY, Chief of Bureau.

EXPERIMENTS ON THE SPOILAGE
OF TOMATO KETCHUP.

BY

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1909.

LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., July 15, 1908.

SIR: I have the honor to submit for your approval a report made by Inspector Bitting of experimental work on the spoilage of tomato ketchup, the conditions contributing thereto, methods of prevention, the action of preservatives, and the length of time that the product will keep under varying conditions of manufacture and temperature, both before and after opening. Every effort has been made to conduct the work in a practical way, and the results obtained can not fail to be of interest and profit both to the manufacturer and consumer. I recommend that this report be published as Bulletin No. 119 of the Bureau of Chemistry.

Respectfully,

H. W. WILEY,
Chief.

HON. JAMES WILSON,
Secretary of Agriculture.

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EXPERIMENTS ON THE SPOILAGE OF TOMATO KETCHUP.

INTRODUCTION.

The tomato, *Lycopersicum esculentum*, is supposed to be native to South or Central America. The large fruits commonly used grow only under cultivation, but the variety with small, spherical fruits, known as *L. cerasiforme*, has been found on the shore of Peru and is considered by De Candolle^a as belonging to the same species as *L. esculentum*. Though grown extensively in Europe, there is nothing to indicate that it was known there before the discovery of America. The tomato was introduced into China and Japan at a comparatively recent date. De Candolle is of the opinion that the tomato was taken to Europe by the Spaniards from Peru and was later introduced into the United States by Europeans. Tomatoes were brought to Salem, Mass., by an Italian painter in 1802,^b who is said to have had difficulty in convincing the people that they were edible. They were used in New Orleans in 1812, though as late as 1835 they were sold by the dozen in Boston. After 1840 they came into general use in the Eastern States, but it was later than this before tomatoes were used freely in the Western States, many persons having the impression that, since they belonged to the nightshade family, they must be unwholesome. The extent to which tomatoes are used at the present time shows how completely this prejudice has been overcome.

The name *Lycopersicum* is from two Greek words, meaning a wolf, and a peach, the application of these terms not being apparent; the name of the species, *esculentum*, is from the Latin, meaning eatable. The common name "tomato" is of South or Central American origin, and is believed to be the term used in an ancient American dialect to designate the plant,^c but its meaning is unknown. The English call the tomato "love apple," which in French is "pomme d'amour."

The tomato is considered a typical berry, the ovary wall, free from the calyx, forming the fleshy pericarp, which incloses chambers filled with a clear matrix containing the seeds. The fruit measures from 1 to 5 inches in diameter, and is red, pink, or yellow when mature.

The plant sports freely, producing many varieties, which differ mainly in the size, shape, and quality of the fruit. The varieties

^a Origin of Cultivated Plants, 1890.

^b Webber, H. J., Yearbook, U. S. Department of Agriculture, 1899.

^c U. S. Dept. Agr., Exper. Sta. Record, 1899-1900, 11: 250.

bearing small fruits are *L. cerasiforme* and *L. pyriforme*, each bearing a two-celled fruit, the former being round, and somewhat larger than a cherry, and the latter pear-shaped. These small tomatoes are used ordinarily for preserves and pickles.

The word "ketchup" is adopted in this bulletin as the form which ought to be given preference. The derivation of the term is not definitely known. The spelling "catchup" given in some of the leading dictionaries appears to be based on the erroneous idea that the first syllable "ketch" is a colloquial form of "catch." Several authorities derive the word from the East Indian or Malayan "kitjap," because "ketchup" was originally a kind of East Indian pickles. Some give the word a Chinese origin, while others assert that it comes from the Japanese. A majority of the manufacturers employ word "catsup," a spelling for which there does not appear to be any warrant.

PROCESS OF MANUFACTURE.

The making of tomato ketchup consists essentially in reducing tomatoes to pulp, removing the skins, seeds, hard parts, and stems, adding salt, sugar, condiments, and vinegar to suit the taste, and



FIG. 1.—A model receiving platform.

cooking to a proper consistency. The methods and practices of the various manufacturers differ, and the difference between the best and the poorest procedure corresponds to that between the best and the worst ketchup. No single factory has all of the best methods at every step of manufacture. Some perform certain details well and are negligent in others. In some, large amounts of money are

spent on equipment to improve a particular point considered advantageous by the trade, while other details essential to the making of a good-keeping ketchup are disregarded. A statement of the best practice as observed at a number of factories, together with some facts obtained from experiments, will be given.

SELECTION AND PREPARATION OF STOCK.

The tomatoes should be home-grown, of a red variety having the minimum of yellow and purple color, be picked when ripe, and delivered to the factory promptly without mashing. All tomatoes should pass over an inspection table, the rotten and otherwise unfit fruit



FIG. 2.—Large receiving room showing the sorting belt.

should be discarded, and the green tomatoes should be returned to crates to ripen. The stems should be removed when the best color is desired, and the tomatoes should be thoroughly washed to remove dirt and mold. Dumping a crate of tomatoes into a hopper of dirty water and playing a gentle spray of water on part of them merely wets the skin and makes them appear bright.

PULPING.

The clean tomatoes should be conveyed to the steaming tanks and subjected to steam heat until the skins burst and the meat softens. After a short heating the tomatoes should be run through a "cyclone" where the skins, seeds, etc., are removed and they are

rubbed to a pulp. To remove very small particles and fiber, the pulp may be run through a sieving machine at once; or, if ketchup of the smoothest possible kind is to be made, this procedure should be delayed until after the cooking. The pulp is collected in a receiving vat, and only such an amount should be provided in advance as will keep the kettles full, as it is better to stop the tomatoes before going to the washer than to have the pulp stand for some hours. In common practice, however, the pulp is either sent to the cooker at once, or it is allowed to stand and partially separate. If tall casks are used for this separation the solids will rise to the top and the clear watery portion is drawn off at the bottom, or the pulp may be strained through cloth bags. The object of this separation is to secure greater concentration of the solids, retain a brighter color, and shorten the time of cooking.

COOKING AND SEASONING.

The cooking may be done in copper kettles, as shown in figure 3, though these are being superseded by enamel tanks containing silver-plated coils in order to secure the brightest color. By using the



FIG. 3.—A section of a kitchen showing the copper cookers.

latter the discoloration due to the splashing of the contents against the walls of the copper vessel is avoided, and economy of space is secured. Whole or ground spices, or acetic acid or oil extracts of the spices may be added to the pulp in such proportion as the particular brand demands. The spices most used are cloves, cinnamon, mace, and cayenne pepper; but paprika, pepper, mustard, cardamon,

coriander, ginger, celery, and allspice are used by some manufacturers. When whole spices are used, it is the practice to suspend them in a cloth bag or a wire basket and to take them out after boiling. They tend to darken the color of the ketchup, a result considered undesirable by some. The ground spices are used sparingly, with the exception of cayenne pepper. The acetic acid extracts of spices are used because they are economical and give a brighter red color than is obtained with the whole spice. The oil extracts produce no discoloration, but they are the most expensive and give an objectionable flavor. Hungarian sweet paprika is now quite largely used and adds to the color as well as to the flavor. Sugar, salt, and vinegar are added in such proportion as may be desired, and in some brands onions and garlic are used.

EVAPORATION AND FINISHING.

The pulp is evaporated rapidly to such consistency as the grade and price will warrant, the reduction in volume being from 40 to 60 per cent. This is accomplished in about forty-five minutes. The cooking is not continued longer than is necessary, as each minute added to the cooking darkens the finished product.

If the pulp has been run through the sieving machine before cooking, the batch may be drawn off into the receiving tank for bottling. If the finishing be done after cooking, the pulp is run into a receiving vat, finished as quickly as possible, and drawn into the tank for bottling. The ketchup may be kept at a high temperature—200° to 206° F.—in the receiving tank by means of a small steam coil, or it may be drawn to the bottling machine through a steam-jacketed tube. Finishing after cooking yields a slightly smoother ketchup than sieving before cooking; but it necessitates handling, reduces the temperature, and increases the chances of infection.

BOTTLING.

The bottles should be thoroughly cleaned as ketchup will not keep if placed in bottles which have been merely rinsed to remove the straw; if the ketchup is not to be given an after process the containers should be sterilized. In the experimental work cork stoppers gave the best results and these should be sterilized in a paraffin bath at 250° F.

PROCESSING.

An after treatment or process is given to bottled goods either in a water or steam bath, the important point being that the center of the bottle be raised to the desired degree of heat. If the ketchup is thin this can be effected quickly, but if it is thick and heavy the heat penetrates the ketchup with surprising slowness.

In a thin ketchup the temperature may be raised from 140° to 190° F. in eighteen minutes or less when the surrounding heat is 195° F.; but in a heavy ketchup it may take an hour or more to accomplish the same result. It is therefore very important that



FIG. 4.—An example of factory practice showing the top row of tanks from which pulp passes by gravity into the cookers, then into the receiver, sieving machine, and final tub ready for the bottling machine or jug filler.

the ketchup be processed immediately after it is corked, before it has time to cool. The rate at which the heating is effected for different goods can be determined by sealing a thermometer in the cork and recording the readings.

CHARACTER OF PRODUCTS.

FIRST-CLASS PRODUCTS.

The factory at which the experiments were conducted has sanitary buildings and surroundings, the floors are of concrete for flushing, and the pipes used in conducting the pulp to the kitchens are porcelain-lined to prevent discoloration from the iron and to insure cleanliness. The tubes which carry the ketchup from the kettles to the receiving tank, finishing machine, and bottler are silver-plated.

Not all of these measures are necessary to make a good ketchup, but they show the care exercised in making an article of good appearance and of the finest quality.

The conditions under which ketchup is made and the care with which the work is done at some of the better factories is equal to that used in the manufacture of any food product. Whole selected fruit is used, cleanliness is maintained at every point, the best grades of spices, vinegar, granulated sugar, and salt are added for flavoring, and the bottles are carefully washed. The ketchup put up under such conditions will have a bright natural color, will remain good as long as the container is unbroken, and will continue in that condition for some time after opening if kept at a fairly cool temperature.

INFERIOR PRODUCTS FROM "TRIMMING STOCK."

In contrast with the strictly high-grade product is the great bulk of the ketchup found on the market. The material is not whole ripe tomatoes, but consists of the waste of the canning factory, commonly designated as "trimming stock," including the green, moldy, broken, rotten, and generally unusable tomatoes, the skins, cores, and stems from the peeling tables, and the surplus juice from the filling machines, all of which may be allowed to stand during the day and be run through the cyclone in the evening. At the end of the season, the frosted and half-ripe fruits may be used. Part of this material can not be considered "sound fruit" as contemplated by the food and drugs act. The pulp is put up in barrels, preserved, and allowed to stand, possibly in the sun, until a sufficient quantity has accumulated for shipment. Old ketchup barrels may be used and be none too clean. As a result, it is not uncommon to see an inch or more of pulp in the bottom of a car at the end of shipment, caused by the blowing out of the barrel heads from fermentation. The sanitary condition of the factory may be poor, the handling of the goods be unclean, the spices be the refuse from the spice houses, the sugar be of the cheapest grade, and the bottles be only rinsed or be used without even that precaution. The ketchup is a concoction so heavily spiced with hot spices that the tomato flavor is lost and might as well be anything else. The color is normally dirty brown.

Between these two extremes are all grades, those for which whole tomatoes, unsorted, are used, those for which trimming stock is worked up promptly during the canning season, and those made from stock of unknown history. Some manufacturers work under good and some under poor sanitary conditions. There can be no doubt that with proper selection and precaution much of the by-product of the canning factory and large quantities of tomatoes which are unsuitable for canning might be used to advantage in the manufacture of ketchup; but it requires a nicety of practice not

generally found at this time. The practice sometimes followed of making some ketchup from whole stock and a large quantity from refuse and using the former for advertising purposes, only serves to emphasize the fact that the goods belong to two distinct classes. One of the uses for a very considerable amount of pulp from refuse stock is the making of sauce for baked beans and other canned goods where the true character can not be observed by the consumer.

During the season tomatoes come in at times in larger quantities than can be made into ketchup promptly. The surplus must be worked up into pulp for storage and may be stored in barrels or in

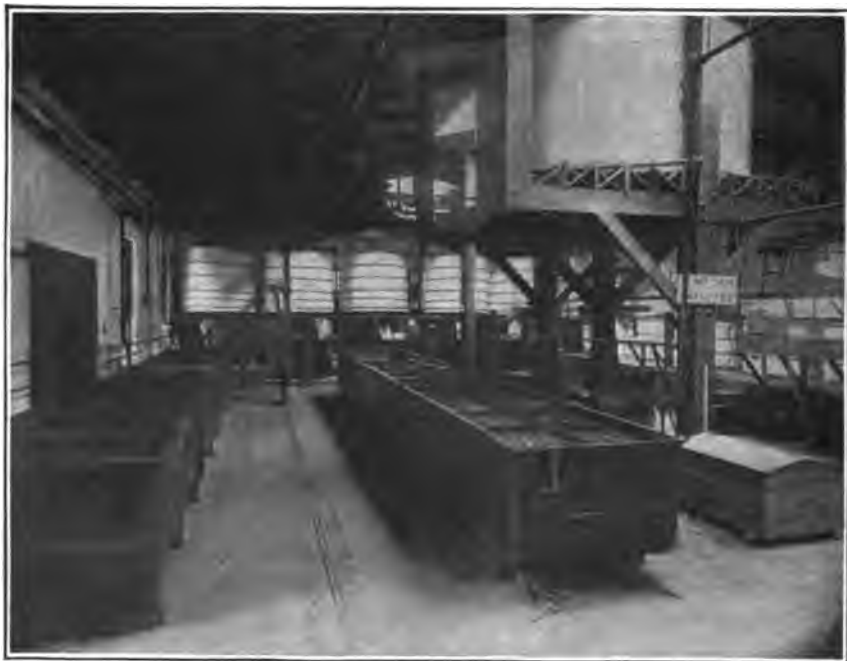


FIG. 5.—Another factory interior, showing large pulp tanks in the rear, cooking tanks on the right, and process tanks in front containing thousands of bottles of ketchup.

tin cans. The pulp stored in barrels will not have as good a color as that put into cans, and the ketchup made from either will not be as bright as that made from whole, fresh stock. The pulp put up in barrels is more liable to spoilage than that put up in cans. The difference in the cost of storage by the two methods is not very great, and some large concerns are using the can exclusively instead of the barrel.

LABELS.

The labels on the ketchup bottles have been improved somewhat in the last year as regards exactness in describing the contents. Formerly, according to the labels, much of the ketchup was made from

whole ripe tomatoes. The question was, What became of the enormous amount of ketchup which it was known had been made from "trimmings?" On this year's ketchup the labels make fewer claims, generally merely stating that it is "tomato ketchup," which is true whether made from whole tomatoes or refuse. The brand is in most cases the guaranty for good quality. It is not safe to judge the quality by the price, for, though usually good quality can not be expected unless the higher price is paid, some of the high-priced ketchup when placed under the microscope has proven to be a very inferior product.

The wide labels on the neck of the bottle are objectionable. Some of these are 2 inches in height, and serve to cover the discolored and spoiled ketchup. As spoilage begins usually in the neck of the bottle, it is difficult to see it when the neck is wrapped with a label, and thus it might easily be overlooked until the main body of the ketchup is affected. The bottles which have the widest labels around the neck are usually the ones provided with one or two large labels on the lower part of the bottle, though some bottles have no other label but the one around the neck. As a rule, however, these are narrow, close to the stopper, and unobjectionable.

In buying ketchup for experimental purposes it was difficult and sometimes impossible to learn its age, as often the grocer does not know it, and at other times he will not tell. It appeared, however, that often the ketchup had been on the grocer's shelf or in the warehouse from one to four years.

MANUFACTURING EXPERIMENTS WITHOUT THE USE OF PRESERVATIVES.

OUTLINE OF THE EXPERIMENTS.

During September, 1907, ketchup was made in experimental batches to determine whether it could be manufactured on a commercial scale without the use of preservatives. These experiments were made to determine (1) the keeping quality before opening the container and (2) the length of time the product will keep without spoilage after the bottle is opened.

The ketchup was made in a factory in which the conditions of manufacture and all the surroundings were sanitary; whole, ripe tomatoes, the same as used in the regular grade of canned goods, were used and the formula and process were for a mild ketchup giving the maximum of tomato flavor. Each batch consisted of 50 gallons of finished goods, from which 1 gross of pint bottles was retained for observation.

The term "regular ketchup" as used in these experiments means the pulp of fully ripe tomatoes, to which was added granulated

sugar, 80-grain, distilled vinegar, table salt, onions, garlic, whole cinnamon, cloves, mace, and ground cayenne pepper. The pulp was cooked in a steam-jacketed copper kettle for forty minutes and reduced about 50 per cent. The finishing was done after cooking. The regular bottles are pint sizes, washed in hot water, rinsed, and then heated to a temperature of 190° F. for thirty minutes or more. The sterile bottles referred to in the experiments were placed in a steam chamber for twenty minutes at 230° F. The corks were sterilized by a bath in paraffin at about 270° F. All of the work was accomplished quickly to insure a smooth, even product with a bright, clean color. Acetic acid extracts and oil extracts of spices were used in such quantities as would give the same amount of spicing as when the whole spices were employed.

In all of the following experiments the ketchups discussed were made in September, 1907, and the last examination reported was made ten months later, in July, 1908:

Experiment No. 1.—Regular ketchup was made, but it was reheated after finishing and bottled in sterile bottles at a temperature of 205° F. No spoilage has occurred at the end of ten months.

Experiment No. 2.—Regular ketchup was made, and it was bottled immediately after finishing in regular bottles at a temperature of 165° F. An after process was given at 190° F. for twenty minutes. No spoilage has occurred after ten months.

Experiment No. 3.—Regular ketchup was made, and was bottled in regular bottles at 165° F., and given a subsequent process at 190° F. for forty minutes. No spoilage has occurred.

Experiment No. 4.—Regular ketchup was made, was bottled in regular bottles at a temperature of 165° F., and given an after process at 212° F. for twenty minutes. No spoilage has occurred.

Experiment No. 5.—Regular ketchup was made, the same being put up in regular bottles at a temperature of 165° F. and given an after process at 212° F. for forty minutes. No spoilage has occurred.

Experiment No. 6.—Ketchup was made in which the acetic acid extracts took the place of whole spices, and the bottling was done at a temperature of 165° F., no after treatment being given. No spoilage has occurred.

Experiment No. 7.—Ketchup was made in which acetic acid extracts were used, and the bottling was done at a temperature of 165° F. in sterile bottles. No after treatment was given and no spoilage has occurred.

Experiment No. 8.—Ketchup was made in which the oil extracts were used instead of regular spices. The bottling was done in regular bottles at a temperature of 165° F., no after treatment being given. No spoilage has occurred.

Experiment No. 9.—Ketchup was made in which oil extracts were used instead of whole spices. The bottling was done at 165° F. in sterile bottles, no after treatment being given. No spoilage has occurred.

Experiment No. 10.—Regular ketchup was made, but the pulp was run through the sieving or finishing machine before instead of after cooking, the object being to determine the effect upon the character of the goods rather than upon the spoilage. This practice could be followed to advantage in making all except the very finest goods, and would give the same condition for bottling as in experiment No. 1.

Experiment No. 11.—Pulp was made in the usual manner and run into barrels while just below the boiling point. The barrels had been thoroughly washed and then steamed for twenty minutes. As soon as the pulp had cooled slightly the bung was

driven in tightly and the barrel was rolled into storage. At the end of sixty days the barrels were opened and the pulp was found to be in good condition.

Experiment No. 12.—Regular ketchup was drawn into 5-gallon jugs which had been sterilized in the same manner as the bottles. These were kept for sixty days and no spoilage occurred.

DISCUSSION OF RESULTS.

Twelve hundred and ninety-six bottles were shipped from Terre Haute to Lafayette, Ind., and some were reshipped in order to duplicate the conditions in trade. Some were kept in a warm temperature and in strong light, others in a comparatively cool place and in the original shipping cases, in order to duplicate the conditions in the warehouse and grocery store. There has been no spoilage after ten months other than that resulting from four or five cork leaks and neck cracks. These experiments have shown conclusively that ketchup can be put up on a commercial scale and delivered to the consumer in perfect condition without the use of a preservative.

It was demonstrated by the first experiment that the goods could be bottled at a high temperature without difficulty, and that subsequent treatment was unnecessary. The after treatment at 190° was tried because it had been found in small experiments that, in giving a higher temperature, the internal pressure would cause more or less breakage of bottles or loosening of corks. After treatment is practiced by some who also use a small quantity of preservative as a further precaution. This treatment is continued from two to three hours at the temperature of high pasteurization.

The process at 212° was given with little breakage, as the bottles used were of good quality. At and above this temperature the breakage may be reduced by either raising the temperature of the ketchup before bottling or applying pressure upon the outside while giving the process.

Neither the acetic acid nor the oil extracts showed any advantage over whole spices in their preservative effects, as all kept. The color was slightly improved, but the flavor was impaired, particularly when the oil extracts were used.

SPOILAGE OF KETCHUP AFTER OPENING.

The question of how long the ketchup should keep after opening the container in order to satisfy the ordinary requirements of consumption was also studied. A local restaurant, serving about two hundred meals and using from one-half to a gallon of ketchup daily, was supplied with the same kind of ketchup used in the experiments, as were also some families. Instructions were given to use the ketchup as they would ordinarily, with the result that none reported any loss from spoilage.

To determine how long the ketchup would keep after opening, 8 bottles from each of the first 9 experiments were kept in the kitchen

at a temperature of about 72° F., 5 were kept in an incubator at a temperature of 95° F., 5 were kept in the laboratory at a temperature of about 67° F., and 4 were kept in an inclosed porch where the temperature ranged from 30° to 60° F. This made a total of 198 bottles. No precautions, other than those of ordinary cleanliness, were taken in opening the bottles, as it was desired to determine the keeping properties under conditions of general usage. The first set of bottles was opened November 5, immediately on being received at the laboratory, all of the ketchup having been kept at the factory until the experiment begun in September was completed. The bottles were covered loosely with a metal cap and observed daily, a record being kept of the date and character of spoilage.

The results showed that the differences in the time and temperature of processing had little, if any, effect in checking the spoilage; neither did the use of acetic acid or oil extracts. The most important precaution in checking the spoilage after opening seems to be to keep the ketchup cool. This is shown by the average number of days which elapsed before spoilage occurred in the sets kept under different temperature conditions. For those kept in the kitchen the average number of days was six, the minimum three, and the maximum eleven. Those in the incubator kept for an average of five days, with a minimum of two days, and a maximum of eight. Those in the laboratory had an average of eight days, the minimum being four days and the maximum twenty-two. Those kept in the porch lasted on an average twenty-seven days, a minimum of twelve days, and a maximum of fifty-eight.

These figures show the definite relation of temperature to spoilage under the conditions of ordinary use. In making the observations, the metal cap was removed each day, but no ketchup was poured off. The spoilage in all cases was due to mold, and usually this formed in the neck of the bottle where the ketchup had splashed, or at the junction of the ketchup with the bottle. The spoilage was recorded as soon as the slightest growth appeared. In actual use if the neck were wiped out when the ketchup had been used and a growth of mold removed on its first appearance with some of the proximate ketchup the time before spoilage occurred could be prolonged. In these experiments the attempt was made to determine how soon growth appeared under the various conditions of temperature named.

The unopened bottles of ketchup were kept in a basement room, the temperature of which is fairly constant, being about 70° F. This is approximately the condition in a grocery where the ketchup is kept on the shelves. Another set of samples from the run of September, 1907, was opened February 11, 1908, to determine if storing in a warm room before opening had any effect on the length of time preceding spoilage. Four bottles were taken from each of the first 9 experiments to make up each of three sets, one of which was kept in

the kitchen, one in the incubator, and one in the porch, making a total of 108 bottles. The average number of days for those kept in the incubator was four, the minimum two, and the maximum six. The average number of days before spoilage in the kitchen was five, the minimum being three and the maximum nine. Those kept in the porch gave an average of twenty-three days, the minimum number being eighteen days and the maximum seventy-three days. Thus it is seen that the ketchup lasted nearly five times as long at a temperature of 30° to 60° F. as it did at 72°; and also that when ketchup is kept in a warm place before opening, spoilage occurs somewhat sooner, the average for the fresh samples opened under the same conditions being one day more with the incubator and kitchen samples and four days more with the porch samples.

A third set of bottles of the ketchup was opened on June 6, 1908, or two hundred and sixty-five days after manufacture. They had been kept in a basement at a temperature of about 70° F.

One set was placed in the incubator at a temperature of 95° F., one set in the kitchen at about 82° F., and one set in the refrigerator at 46° F. The weather was warm and the conditions favorable to the spoilage of fresh foods. The minimum time for spoilage in the incubator was two days, the maximum time four days, and the average time three and two-tenths days. The minimum time in the kitchen was two days, the maximum time six days, and the average time four and four-tenths days. The minimum time in the refrigerator was nine days, the maximum time nineteen days, and the average time thirteen and sixty-six one-hundredths days.

These data are grouped in the following table for easier comparison:

Time of spoilage of ketchup at different temperatures after opening.

OPENED ON NOVEMBER 5, 1907, IMMEDIATELY UPON RECEIPT FROM FACTORY;
MAXIMUM AGE, FIVE WEEKS.

Place of storage.	Temperature.	Lapse of time before spoilage.		
		Average.	Minimum.	Maximum.
	° F.	Days.	Days.	Days.
Incubator.....	95	5	2	8
Kitchen.....	72	6	3	11
Laboratory.....	67	8	4	22
Porch.....	30-60	27	12	58

KEPT AT 70° F. FOR ONE HUNDRED AND FIFTY DAYS BEFORE OPENING ON
FEBRUARY 11.

Incubator.....	95	4	2	6
Kitchen.....	72	5	3	9
Porch.....	30-60	23	18	73

KEPT AT 70° F. FOR TWO HUNDRED AND SIXTY-FIVE DAYS BEFORE OPENING ON
JUNE 6.

Incubator.....	95	3.2	2	4
Kitchen.....	82	4.4	2	6
Refrigerator.....	46	13.66	9	19

SPOILAGE OF UNOPENED KETCHUP.

Another test was made to determine whether the ketchup would spoil when kept in a warm place, but not opened. Three bottles from each experimental batch were placed in the incubator November 7, 1907, and were kept there until December 23, 1907—forty-six days—and in that time there was no sign of spoilage. They were then opened and kept in the laboratory; the average number of days before spoilage occurred is indicated in the following table:

Average number of days before spoilage of ketchup after opening (kept 46 days at 95° before opening).

Experiment No.	Days before spoilage.	Experiment No.	Days before spoilage.
1.....	2½	6.....	4½
2.....	4½	7.....	4½
3.....	3½	8.....	4½
4.....	5	9.....	3½
5.....	5½		

It will be observed that these samples spoiled in about the same length of time as the bottles opened in February and tested in the incubator, so that similar results were obtained by keeping unopened ketchup one and one-half months at 95° F. and keeping it five months at 70° F. From the results of the experiments it is evident that the ingredients of the ketchup in the proportions used are not antiseptic, and it is also apparent from the number of organisms found and the rapidity of their multiplication that ketchup is a good, nutritive medium. Yeasts and molds are the predominating organisms, and, as the ketchup is acid and also contains sugar, and these organisms are found on tomatoes in the field, their predominance in the ketchup is explained.

SPOILAGE OF MARKET BRANDS.

To determine the keeping properties of the ketchup on the market, various brands were obtained from the grocery stores. In the majority of cases nothing was known of the ingredients or methods of manufacture, except what appeared on the labels. No date of manufacture was given, and in some cases the dealers did not know the age of the product.

There were 104 bottles of ketchup opened to find out how long they would remain in good condition. These were kept in the laboratory, though the temperature was higher than that at which ketchup should be held. Of the 104 bottles there were 66 without preservative, according to the labels, 46 of which spoiled. Of the 20 which did not spoil, 2 formed crystals of benzoic acid on the covers of glass dishes

during evaporation. Of the 39 which, according to the labels, contained sodium benzoate, 15 spoiled. The bottles of unspoiled ketchup after remaining in the laboratory for about a month were placed in the incubator at 95° F. for three weeks, and were then taken out, and have been left in the laboratory since. The metal cap had been taken off frequently for observation, and the ketchup exposed, but the treatment did not cause them to spoil.

The average number of days after which spoilage occurred for the 46 bottles without preservative was about fifteen, the minimum number being four days, the maximum number ninety-four days. The average number of days preceding spoilage in the case of 15 bottles with preservative was twenty-four days, the minimum number being three and the maximum sixty days. The majority of these had 0.1 per cent of sodium benzoate present; the others had a smaller amount, according to the manufacturer's label. These data are not at all conclusive and further work on material of known history will be necessary.

STERILITY OF KETCHUP.

To determine the sterility of ketchup, cultures were made from 77 of the bottles. The method used was to wipe the bottles and cork stoppers with a damp towel and then remove the cork. The cork puller which was used grasps the neck of the bottle in such a way as to cover the opening and remove the cork without the inrush of air that occurs when the ordinary corkscrew is used. A flame was then passed over the mouth of the bottle, after which the upper layer of ketchup was poured out, so as to discard any material which might have been contaminated in handling. Tomato gelatin was used as a medium and cultures were made in petri dishes.

There were 17 plates on which no organisms developed, indicating that the ketchup was sterile. Of the 60 plates having organisms, 54 had molds, 22 of these having molds alone; 21 plates had yeast-like organisms, 3 plates having these only; 29 plates had bacteria, 4 having bacteria alone. Sometimes a plate would have only one form of organism, but more often there was a mixture present. Of 15 plates having only one form of organism, 3 had yeast alone, 2 bacteria alone, and 10 had mold alone. Of the 77 bottles of ketchup from which the inoculations were made, 41 were without and 36 with preservative, and of the 17 sterile ketchups, 8 contained sodium benzoate and 9 were without preservative.

A considerable part of the experimental ketchup proved not to be sterile. The organisms present were of the class which require oxygen for their growth and therefore they had only been arrested in their activity. No growth could take place so long as

the air was excluded and therefore no spoilage could occur. When the cork was drawn, the organisms could grow and cause spoilage, and this is a much more potent factor than the entrance of germs from without. Bottling and sealing the ketchup quickly while hot so completely excludes the air that only a few colonies of yeast or mold may be found on subsequent microscopical examination. Filling at a low temperature and corking while cool allows sufficient air to remain incorporated in the ketchup and neck of the bottle to permit a considerable growth of the organisms and a product derived from good stock may thus acquire the appearance of ketchup derived from partially decayed material. A ketchup in which bubbles of air are incorporated in filling may show a growth of mold at each bubble throughout the mass. The foregoing statements apply to ketchup containing sodium benzoate as well as to the non-preservative goods of the character used in these experiments.

EXPERIMENTS WITH PRESERVATIVES.

SODIUM BENZOATE.

The preservative in general use in ketchup is sodium benzoate. Salicylic acid is used, but only to a limited extent. The amount of sodium benzoate used, according to the labels, varies from one-sixteenth to one-tenth of 1 per cent; but on some labels the amount is not stated. Experiments were made to determine the amount necessary to check the spoilage of ketchup.

Two organisms, a mold and a yeast, were selected on which to make the tests. The mold was the ordinary blue mold, *Penicillium*, which was present in many of the brands of ketchup and is found commonly on acid foods. It was selected on account of its prevalence and resistive power. The yeast was obtained from ketchup and was also a vigorous grower, forming a thick, wrinkled film on various media. Any effect on the growth of the yeast could be seen readily in its manner of forming the film.

Portions of tomato gelatin to which 0.1, 0.5, 1, and 2 per cent, respectively, of sodium benzoate were added, were first inoculated with the mold. There was no development in those containing 1 and 2 per cent; a retarded development resulted in that containing 0.5 per cent, and the growth when 0.1 per cent was used was nearly normal, showing very little difference from that in the gelatin without sodium benzoate.

Ketchup was next tried as a medium, but the amount of benzoate was reduced to one-sixteenth, one-twelfth, and one-tenth of 1 per cent, as it was thought that some of the other constituents of the ketchup were antiseptic to a slight degree. The growth in the ketchup was

irregular, though the benzoate checked development in all. Equal amounts of benzoate were used in tomato bouillon, with practically the same results as in the ketchup. The development was checked in all, and in some plates one-sixteenth of 1 per cent seemed to be fully as efficacious as one-tenth of 1 per cent. When the mold was examined under the microscope, the filaments were found to be much swollen and distorted in shape, and filled with a coarsely granular protoplasm, containing much fat, as indicated by the blackening with osmic acid. The culture containing the mold which gave the least development seemed to show the least distortion and swelling of the filaments.

The results indicated that in using sodium benzoate as a preservative there is uncertainty as to results, even when using the maximum amount allowed—one-tenth of 1 per cent. They also indicated that this preservative had an injurious effect on the living matter of the mold. (See Pl. II; compare with normal growth, Pl. I.)

SALT.

The effect of salt in checking development was tested by using tomato bouillon as a medium and adding 5, 10, 15, 20, 25, and 30 grams of salt, respectively, to 100 cc. These were inoculated with the mold. The 5-gram solution seemed to have no effect on development. When 10 grams were used growth appeared as soon as in the bouillon without salt, but was not so extensive. In the 15-gram solution growth was retarded four days, and most of that which did develop remained submerged, the mold growing normally on the surface. With 20 grams the growth was five days slower than the normal in starting, and after that there was only a slight development. In the 25-gram solution, the growth started at the same time as when 20 grams were employed, but remained stationary, while with the 30-gram solution, no development occurred.

The yeast was checked slightly by 5 grams, and very materially by the 10-gram solution, as it required two days for a thin, delicate film to form, whereas in ordinary solutions a rather thick film is formed within twenty-four hours or even in less time. There was no development in the 15-gram solution.

SUGAR.

The effect of sugar was tested on both the mold and the yeast by adding it to tomato bouillon. It was supposed that a low percentage of sugar like the salt would plasmolyze the cells, and in this way check growth, but it seemed to have no effect until the amount was increased to 25 grams per 100 cc of bouillon. In this solution growth appeared as soon as with the weaker solutions, but there was a smaller amount. In the 25 to 40 gram solutions there was less development as the

amount of sugar increased. In the 70 and 75 gram solutions growth was delayed one day in its appearance. In the 80, 85, and 90 gram solutions growth was delayed two days, the colonies growing submerged at first, but after a time forming on the surface. The mycelium remained very thin, but a thick layer of spores formed. From this point on the amounts were increased by 10 grams up to 200. The development became slower and less successively until 170 grams were added. In this case a small colony appeared on the surface in seven days, but seemed to grow less after that. The solutions were held, and in time crystals separated from the thick sirups. After two months dry-looking colonies developed along the edges, forming a ring, and some formed on the surface, these occurring also in the flasks containing 170, 180, 190, and 200 grams of sugar per 100 cc. The colonies were a dull greenish drab in spots, the remainder being white.

For the yeast the 80-gram solution of sugar was the strongest in which any development took place.

SPICES.

Experiments to determine the value of the spices as antiseptics were made, using water infusions, acetic-acid extracts, and oil extracts.

WATER INFUSIONS.

In making the water infusions 20 grams of the whole spices, with 200 cc of water, were boiled for forty-five minutes. This is approximately the length of time that the spices are cooked in the ketchup in the factory. The liquid was then filtered and from 0.1 to 5 cc of the filtrate was used in 10 cc of tomato bouillon. The same organisms were used as in the former experiments.

The tests showed that cinnamon and cloves were the strongest antiseptically. These checked growth when used in small amounts, but it required 3 cc of the cinnamon and 1 cc of the cloves to inhibit the growth of the mold. Mustard, paprika, and cayenne pepper checked growth also, but 5 cc, the highest strength used, did not inhibit growth. The ginger, mace, and black pepper had no apparent effect in the quantities used.

The effect of the spices on the development of the yeast was somewhat different from their effect on *Penicillium*. The cinnamon showed the strongest action, 3 cc being effective, whereas 5 cc of the cloves was required. The cayenne pepper came next in effectiveness, and after that the black pepper. The ginger, mace, and mustard solutions had no effect in the strengths used.

The remainder of the spice infusions were kept in glass-stoppered bottles in the laboratory, and in a few weeks' time there was a coating of mold formed over the surface of the mace, the mustard, and

the black and cayenne peppers. The paprika had small, stunted colonies dotting the surface.

At the time that these experiments were made a quantity of the ground spices were placed in large petri dishes and water was added to make a heavy paste. One set of these was inoculated with the mold, and another set with the yeast, and all were kept in a warm place. No development of either organism appeared on the cinnamon, cloves, or mustard; on the others a growth first showed in three days. On a normal medium growth appears in twenty-four hours. On the mace, paprika, and cayenne pepper the *Penicillium* and yeast with which the pastes were inoculated were overgrown in a few days with black mold (*Rhizopus nigricans*).

ACETIC-ACID EXTRACTS.

In the manufacture of ketchup acetic-acid extracts of the spices are sometimes used instead of the whole spices, on account of their supposed antiseptic properties as well as their greater strength and convenience in handling. One minim of the standard acetic-acid extracts is equal in strength to 1 grain of the whole spices. The acid extracts obtained included allspice, celery, cloves, coriander, garlic, and black pepper.

In the tests 0.1, 0.2, 0.3, 0.4, 0.5, and 1 cc, respectively, of the extract was added to 10 cc of tomato bouillon. One set was inoculated with the mold and another set with the yeast. In the case of the mold, no growth occurred with the allspice and cloves; the celery checked the growth materially, there being no indication of mold until the sixth day. Normally a fairly strong growth occurs in twenty-four hours. In the solution containing 0.3 cc there was only one small colony in thirteen days, and no further development. In the solution containing the coriander, the growth in the 0.5 cc solution did not appear for three days, the 1 cc solution showing no growth. The garlic had practically the same effect as the coriander, while the black pepper was stronger, no growth appearing in the solution containing 0.5 cc.

The yeast was slightly stronger in resisting the effect of the extracts. No growth appeared with the allspice and cloves; 0.5 cc of the celery and 1 cc of the coriander were required to inhibit growth, and the garlic and black pepper gave similar results, a weak development occurring in the solutions containing 1 cc.

OIL EXTRACTS.

Oil extracts of the spices were tested in the same manner as the water infusions and the acetic-acid extracts. The oils were so strong that in order to handle them easily they were mixed with equal volumes of alcohol, except that the mace, which was in the form of a

paste, was mixed with two-thirds its volume of alcohol. To 10 cc of tomato bouillon were added 0.1, 0.2, 0.3, 0.4, and 0.5 cc, respectively, of the oils of cinnamon, cloves, mace, mustard, and black pepper.

In the case of the mold, there was no development in the solutions containing cinnamon, cloves, and mustard; in those containing mace and black pepper the development was slower than the normal, that in the black pepper being more pronounced. On the yeast the effect was similar, no development occurring in the cinnamon, cloves, and mustard, and a retarded development taking place in the mace and black pepper, that in the black pepper being the more pronounced.

The experiments show that some of the spices, notably allspice, cinnamon, and cloves have decided antiseptic value, but that the peppers are not as valuable as is generally supposed.

The oil extracts have been advocated for use in ketchup instead of the whole spices, but in quantities which would be useful antiseptically their use would be objectionable, for when present in approximately the same proportions as are the whole-spice infusions, the flavor is too strong and masks the more delicate flavor of the tomato. The acetic-acid extracts are more effective than are the water infusions, and they are not objectionable in the ketchup.

VINEGAR AND ACETIC ACID.

An experiment was made to determine the antiseptic value of vinegar and acetic acid. Commercial 50-grain distilled vinegar was used. It was found that when 30 per cent of this vinegar was added to the tomato bouillon the development of mold was checked and the extent to which it was checked increased with the increased amounts of vinegar. The development in the solution containing 30 per cent of the vinegar was two days later than the normal in starting, while the solution containing 100 per cent was eleven days delayed and showed but little growth.

An 80 per cent solution of glacial acetic acid was used. One-half of 1 per cent added to the tomato bouillon checked growth to the same extent as 30 per cent of vinegar, and no development occurred when the quantity was increased to 2 per cent.

Experiments were then made in which vinegar was added to the ketchup in proportions varying from 1 part in 32 to 1 part in 8, with the result of greatly delaying the appearance of the mold as the proportion increased. With the increase in vinegar it was necessary to add sugar and slightly more spices to overcome the pungency of the acid and thus insure good flavor. The addition of the vinegar to the pulp had the effect of arresting the action of the oxidase and thus the bright color was maintained.

The usual custom in factory practice is to add the vinegar near the close of the cooking process otherwise a considerable portion

of the acid will be driven off. This practice was followed in the experimental work, but it has since been found that continued heating in the presence of the acid has some effect upon sterilization, and therefore the increased amount of vinegar is effective not only because of the additional acid present, but also because the heating in the after process is thereby rendered more efficacious.

This line of experiments gives promise of practical results in producing a ketchup which will not only keep while in the bottle, but will also keep longer after it is opened. Each manufacturer must work out the quantities that could be used with his formula and still retain the character of his goods.

OIL.

In ketchup manufacturing it is customary, if an agitator is not used, to put a small amount of fat in the kettle to check the ebullition during the reduction of the pulp. The amount used in this manner is not sufficient, however, to be apparent in the ketchup. Brannt^a states that in some factories, where the trimmings are allowed to accumulate for the season, they are given liberal doses of oils and condiments when cooked, in order to disguise their defects, so that the product can be placed on the market as "fresh tomato catchup." That the use of oils is increasing is evident from the comparison of the ketchup of the past season with that of former years.

When oil is used in ketchup, it is easily detected under the microscope, as it appears in the form of shining, yellow globules which blacken gradually when treated with osmic acid. Besides this, the oil comes to the surface of the ketchup, where it can be seen readily, and if considerable oil has been used a distinct layer is formed. When the ketchup has been made for some time, the oil changes so that the ketchup has a peculiar "greasy" odor, or the oil may be so changed as to give a decidedly rancid smell to the ketchup. Oil usually causes a deterioration in flavor and odor, though some of the ketchups to which it has been added do not spoil readily. Olive oil, cottonseed oil, and oleomargarine are used. That the oil is not considered one of the regular known ingredients of the ketchup is shown by the failure to declare its presence on the label.

To test the antiseptic value of oils in ketchup, experiments were made, using olive oil, cottonseed oil, and oleomargarine in the proportions of 1 part of oil to 1,000, 750, and 500 parts of ketchup, respectively. The ketchup was made in small quantities, 2 gallons for each experiment. After bottling, all except the check bottles were inoculated with *Penicillium* and kept at kitchen temperature. All spoiled, and neither the quantity nor kind of oil used had any

^a Brannt, W. L., *A Practical Treatise on the Manufacture of Vinegar*, 1900, p. 455.

marked effect in preventing spoilage. That the oils affected the development of the mold was evident. The mold developed first at the junction of the ketchup with the bottle forming a ring which spread gradually over the surface developing a somewhat heavy mycelium. This remained white longer than usual, spores forming very gradually, as indicated by the change in color from white to a delicate blue. At the end of three weeks only spots of color appeared on the surface and these were still blue, though in ordinary development the blue color changes to green in two or three days.

Another test was made, using olive oil only, and in the proportions of 1 part of oil to 500, 400, and 300 parts, respectively, of the ketchup. Reduction was made in a steam-jacketed kettle, the oil being added when the ebullition of the ketchup was the strongest, after which the boiling was continued for fifteen minutes. The ketchup was bottled, unsterilized bottles being used, then covered loosely with the metal caps.

The time required for the ketchup to spoil was longer than in the first set, but there was not sufficient difference nor enough uniformity in the time to indicate that the use of oil in ketchup is desirable, even if the change of flavor and odor be not taken into consideration. The average number of days before spoilage for those containing 1 part of oil to 500 parts of ketchup, was thirteen and two-thirds days; one has has not yet spoiled (a period of forty-five days), while the first bottle spoiled in four days. Those having 1 part of oil to 400 parts of ketchup had an average life of nine and three-fourths days, the minimum being three days, and the maximum twenty-six days. Those having 1 part to 300 parts of ketchup on an average did not spoil for six and three-fourths days, the minimum being four days, and the maximum eleven days.

The failure of some of the bottles to spoil, though similar in every known respect to those which did spoil, is a feature peculiar to ketchup and is familiar to manufacturers who make careful tests before putting their product on the market. For this reason a rather large number of bottles should be used in a test in order that the results may be approximately accurate and represent general conditions.

STUDY OF *PENICILLIUM* IN KETCHUP.

Penicillium is a plant which is distributed widely and apparently is able to grow wherever organic matter is found, though flourishing best when the material contains acid. It causes loss in canneries, breweries, distilleries, etc., the only use made of it being in the manufacture of Roquefort cheese, the immature cheese being inoculated with the conidia for the effect the mold produces in the maturing process.

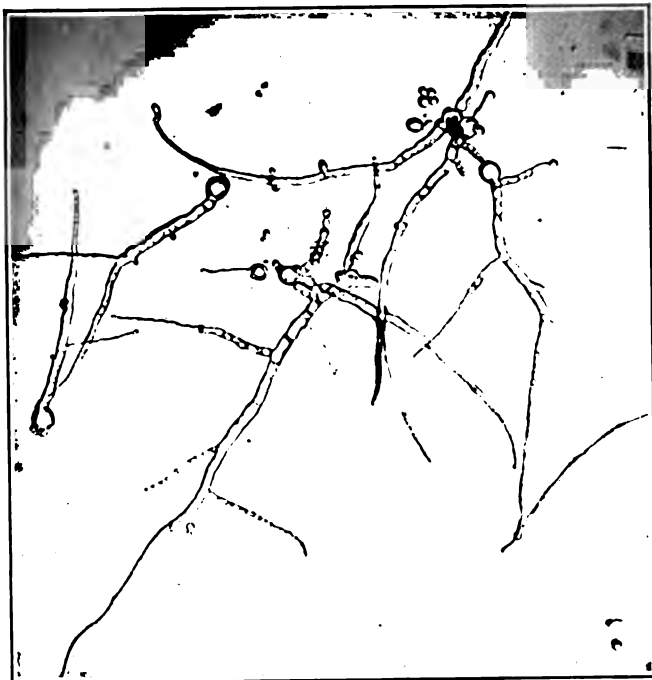


FIG. 1.—CONIDIA, NORMAL SIZE AND IN VARIOUS STAGES OF GERMINATION, SOME WITH BRANCHING HYPHÆ (X 325).

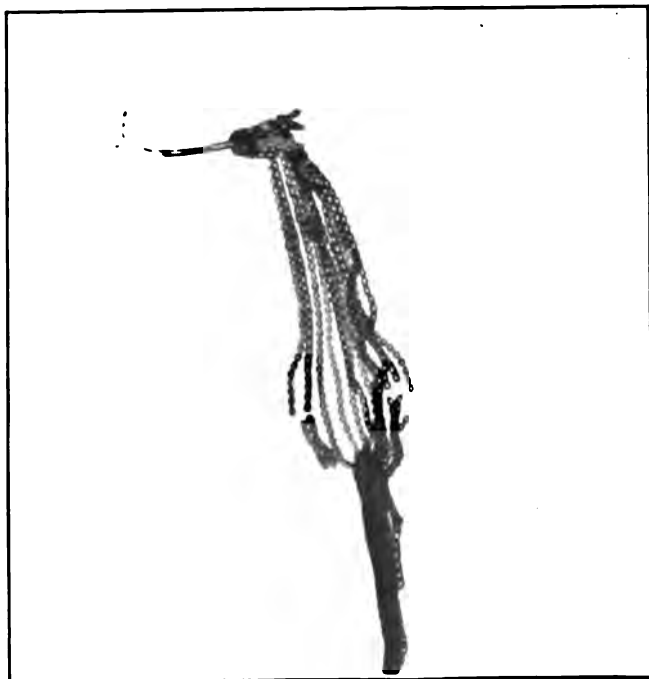


FIG. 2.—CONIDIOPHORE, SHOWING UNUSUALLY LARGE DEVELOPMENT OF CONIDIA, FROM CULTURE IN MOIST CHAMBER (X 325).

PENICILLIUM.

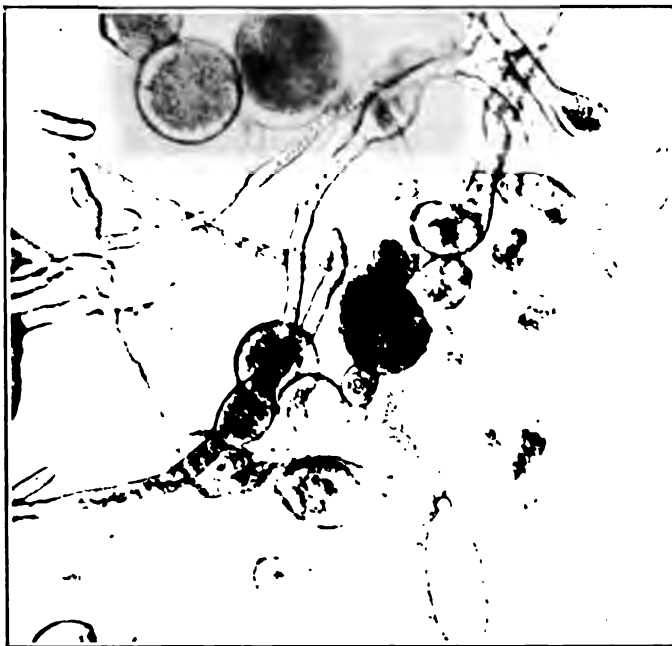


FIG. 1.—CONIDIA AND HYPHÆ FROM CULTURE IN EXPERIMENTAL KETCHUP CONTAINING ONE-SIXTEENTH OF ONE PER CENT OF SODIUM BENZOATE (X 325).



FIG. 2.—CONIDIA AND HYPHÆ FROM CULTURES IN EXPERIMENTAL KETCHUP CONTAINING ONE-TENTH OF ONE PER CENT OF SODIUM BENZOATE (X 325).

CULTURES FROM KETCHUP PRESERVED WITH SODIUM BENZOATE.

DEVELOPMENT.

In developing, the mold forms a white felt-like mass, covering the medium on which it is growing; then as development proceeds, it changes to bluish-green, and finally to a darker, duller color. The change in color is accompanied by a change in structure, the surface becoming powdery in appearance, a slight current of air being sufficient to dislodge a cloud of fine dust. This fine dust is formed of small, spherical bodies, the spores or conidia (from the Greek meaning *dust*). These need no resting period, but are able to develop at once. When the conidia lodge on a moist substance they swell to a much greater size and then send out a tube from some part of their surface. The tube lengthens and septa form, dividing the tube into sections, or cells. At the same time branches are sent out, which again form other branches. The original conidium sends out a second branch shortly after the first one, and usually from the opposite side, and may even send out a third one. The formation of the septa and the subbranching goes on in all, so that in a short time the branches mat together and form a felt-like cover.

REPRODUCTION.

After a shorter or longer period of development, dependent on the conditions, branches are sent perpendicularly from the substratum, and into the air. These branches cease their growth in length, sending out branches near the tip, which take the same general direction as the original branch. Each of these subbranches is called a sterigma (from the Greek word meaning *support*). In vigorous development the sterigmata may form secondary branches, the whole forming a tassel-like arrangement. The tip of a sterigma enlarges, a septum forms around the enlargement, cutting it off from the sterigma, and forming a conidium. The sterigma develops to the original length and another conidium is formed, the operation being repeated many times, thus forming a chain of spores. As the other sterigmata are also forming conidia in the same manner, a series of these chains is formed close together. After the cessation of conidial development, the filament below the sterigmata is disorganized, setting free the conidia. The filament and head together are called the conidiophore (Greek, dust-bearer).

Penicillium forms spores sexually, but the conditions for their formation are unknown. Brefeld obtained them by growing the mold on damp bread placed between two glass plates, and excluding the air. Lindner obtained carpospores on a wort gelatin culture in a petri dish, from which the air was excluded. The writer has tried various methods for obtaining carpospores, but so far without success. Moist chambers were used with various media, excluding the air.

The development of the mold is seemingly dependent on the amount of air in the chamber at the time of sealing. After the air is exhausted, the conidiophores assume fantastic forms, developing only one or a few sterigmata, and on these one or few conidia. In other cases the conidiophores are fascicled, in no cases, however, forming the conidia as luxuriantly as when air is supplied. The hyphæ become clear, much vacuolated, and develop more septa, and some of the cells become much enlarged. An enlarged cell will often contain two or three septa, thus forming cells that are not larger than disks. In cultures from which the air was excluded from the start, no development took place. In test-tube cultures sealed with paraffin after twenty-four hours, the mold developed on the surface of the gelatin, forming a felted white mass, but no conidia nor carpospores were formed.

GROWTH IN KETCHUP.

The form of *Penicillium* which was used in the experiments was isolated from ketchup in which it grew luxuriantly. When conidia are first formed on the ketchup, they are a delicate blue in color; they then become bluish green, then green, and finally olive. The development of the color of mold growing on ketchup is practically the same as when grown in wort, tomato bouillon, pea bouillon, or gelatin made with these solutions as a basis. In ketchup containing sodium benzoate, the blue color appearing first remains for a long time, and in old cultures the mold is a dull drab, not olive, as in normal development.

In ordinary ketchup made without a preservative, the mold forms a heavy, wrinkled mycelium, showing a large development of conidia. In the bottles of ketchup, the mold pushes down into the ketchup, becoming entirely submerged, a clear liquid covering the mold and separating it from the ketchup. This occurred in more than one hundred bottles. No secondary mycelium formed on the surface of the liquid, a method of development which frequently occurs in ordinary media when a mass of mold is submerged.

An exception to this was shown in ketchup which had developed the mold in the laboratory. The bottles were then put in the refrigerator for two weeks. During this time scarcely any development took place; but after they were again placed in the laboratory, the mycelium pushed down into the ketchup and a new, very thin mycelium developed on the surface. The filaments when seen under the microscope were swollen, had irregular outlines, and a comparatively smaller number of septa, and were filled with a coarsely granular protoplasm. The ends were blunt and misshapen and the sterigmata were irregular, tending more toward a fasciculated arrangement, and forming fewer conidia. The filaments from the vinegar and acetic

acid media had the same appearance as those developed on ketchup, but had a smoother outline.

TEMPERATURE TESTS.

The limits for the germination of *Penicillium*, as given by W. J. Sykes,^a are 2° to 43° C. (35° to 110° F.), and the most favorable temperature 22° to 26° C. (72° to 79° F.). This author states also that according to Pasteur the dry spores retained their vitality at 108° C. (226° F.), but that they were soon killed when immersed in boiling water. Klöcker^b quotes Pasteur as saying that the conidia are killed if exposed to a temperature of 127° to 132° C. for half an hour, but that they retain life at 119° to 121° C.

A series of tests was made to determine the thermal death point of the moist and dry conidia of the *Penicillium* used in the experiments, a young, vigorous development on ketchup being used. The flasks were kept under observation for a month after the tests were made, as in many cases a development does not occur in the usual time. The high temperatures applied for longer periods of time were tried first, but both temperature and time were reduced as results from the series were obtained. Only the conditions obtaining in the final tests are given in the table. It was found that the *Penicillium* used did not have the high resistance supposed.

The tests were made in small flat-bottomed 10-cc flasks, tomato bouillon being used for the tests on moist conidia. The bouillon was used so as to have the conidia in a nutritive medium after the test was made, without transferring. The time for those at 100° C. was estimated from the time of ebullition. At the end of the specified time, the flasks were cooled promptly under running water. As the flat bottoms gave comparatively large surface, the heating and the cooling could be effected in a short time. For the tests below 100° C. a vessel of water was heated to the desired temperature, and the flasks were immersed in it and shaken constantly. The dry conidia were placed in test tubes which were immersed in boiling water for the desired time and cooled under running water, after which 10 cc of sterilized tomato bouillon was added. After determining the death point in this manner and finding it to be much lower than had been supposed, it was decided to make the test again, but using ketchup as the medium. Ten grams of ketchup were sterilized, then inoculated from a vigorous growth of mold, and tested with a set in which the tomato bouillon was used. For those below 100° C. the two flasks which were to receive the same temperature were held in the vessel of water at the same time, so that

^a Principles and Practice of Brewing, 1907, p. 284.

^b Ibid., p. 281.

32 EXPERIMENTS ON THE SPOILAGE OF TOMATO KETCHUP.

as nearly as possible the treatment would be identical. The following results were obtained:

Thermal death point of moist and dry conidia of Penicillium.

PENICILLIUM IN 10 CC OF TOMATO BOUILLON.

No. of experiment.	Temperature.	Time of heating.	Time before germination.	Period of observation and developments.
	° C.	Minutes.	Days.	
1.....	85	1	3	Dark strings from spores; 9 days; no development.
2.....	80	1	3	
3.....	75	1	3	
4.....	70	5	3	
5.....	65	5	3	Dark strings running from spores; 9 days; growth normal, spots on surface. Do. Do.
6.....	60	5	3	
7.....	55	5	3	

PENICILLIUM IN 10 CC OF KETCHUP.

1.....	100	3		
2.....	100	2		
3.....	100	1		
4.....	100	1		
5.....	100	Instant.		
6.....	85	1	2	Colonies on sides; 8 days; surface covered. green.
7.....	80	1	2	Do.
8.....	75	1	2	Do.
9.....	70	5	8	Colony on surface.
10.....	65	5	9	Do.
11.....	60	5	3	Colonies on sides; 8 days; surface covered, green.
12.....	55	5	4	Do.
13.....			2	Ring around sides; 3 days; surface nearly covered.

DRY CONIDIA.

1.....	100	10	4	Rough appearance, like that in ketchup.
2.....	100	15	4	Do.
3.....	100	20	7	Slight growth.
4.....	100	25	10	Growth barely perceptible.
5.....	100	30	10	Do.
6.....	100	35		Conidia stained readily, showing they were dead.

YEAST.

1.....	55	5	2	Wrinkled film; liquid turbid.
2.....	60	5	2	Do.
3.....	65	5	2	Thin, smooth film; liquid clear.
4.....	70	5		
5.....	75	5		
6.....	100	Instant.		

The moist heat was very effective in destroying the vitality of the conidia of *Penicillium*, the death point being 27° C. higher than the maximum temperature for germination as given by Sykes. The heating was more effective in destroying germs when applied to bouillon than to ketchup, no development taking place for any temperature above 65° C., even when applied for a short time.

In the ketchup the lower temperatures for the longer periods of time were more effective in checking the development, even though they did not destroy the vitality. In the ketchup, with the exception of Nos. 9 and 10, the colonies started invariably along the sides of the flasks. The greater access of air to those on the sides would

account for this. The conidia on the sides of flasks Nos. 9 and 10 must have been destroyed, as no development took place in either case except in the center of the surface.

The dry conidia were destroyed at 100° C. when heated for thirty-five minutes; they did not reach a normal development in any case, even when heated for only ten minutes, many of the conidia being destroyed by this treatment. Where development failed to take place, the conidia were stained with a water solution of eosin, so as to be sure that the effect was death, and not an arrested development.

The results of the tests do not agree with those obtained in factory practice, where the ketchup is cooked at 100° C. for at least forty minutes and sometimes for fifty or fifty-five minutes, depending on the consistency of the pulp.

HISTOLOGICAL STRUCTURE OF KETCHUP.

In ketchup are found parts of all the various tissues of the tomato broken into fine pieces by the action of the cyclone. Although the sieves take out the seeds, skins, and any large pieces, particles of the various tissues are present in size sufficient for identification. Among the distinctive features are the red crystalline bodies in the parenchyma, which serve to a certain extent to distinguish the parenchyma from that of other plants which might be used for adulteration, and serve also to differentiate the natural from the artificially colored ketchup. Some of the red dye used colors all protoplasm indiscriminately, even that of the fungi present, and as a colored ketchup is usually poor stuff, containing many fungi, the mold filaments, yeast cells, and bacteria receive their share of the color. Other red dye used is in the form of fine powder, which does not go into solution, but is distributed as irregular particles which are distinct from the red crystalline bodies.

Good ketchup made from whole tomatoes has a clean appearance readily distinguishable under the microscope; but the poor ketchup has usually a superabundance of fungi present, fully developed colonies of mold, many forms of conidia, besides yeast-like cells, and different forms of bacteria. All of these may be dead, but neither preservatives nor dosage of odorous spices can disguise their presence. In some of the ketchup examined, which was put up in attractive form and labeled as being made from the whole tomatoes, and which had the appearance and odor of good ketchup, the microscope showed the presence of such quantities of fungi as to leave no doubt that the tomatoes were spoiled when cooked. It is presumable that some of the dealers placing this sort of stuff on the market do not know its condition themselves, and either buy their pulp from other factories or trust its manufacture

to employees whose only care is that the ketchup shall have a bright color and shall "keep." Some of the mould filaments and conidia are distorted in the same way as those of the *Penicillium* are when grown in ketchup to which sodium benzoate has been added.

The ketchup made from sound tomatoes and manufactured in a cleanly manner has practically no fungi present. The ketchup that was used in these experiments was made at different times during the season and was of this character, no bottle examined showing mold filaments when first opened.

MICROSCOPIC EXAMINATION OF SOME COMMERCIAL BRANDS.

In examining ketchup the color, odor, amount of discoloration, presence of foreign tissue, foreign coloring matter, oil, and fungi were determined. If no preservative was mentioned, some of the ketchup was put in petri dishes and inoculated with *Penicillium* to determine whether growth could take place. The following examinations are reported, as they represent some of the best known brands on the market:

No. 9.—Opened September 2, 1907; age unknown; pint bottle; no preservative mentioned; not spoiled July 6 of following year. This ketchup was guaranteed to be made from fresh, ripe, tomatoes by a new process. The color is an unnatural red, has not faded, and the odor is good. The microscope showed the presence of much refuse, and large quantities of fungi, whole colonies of molds, the filaments distorted, many yeast cells, and bacteria. The red color was not confined to the red crystalline bodies, as is the case in ripe tomatoes, but the whole of the protoplasm of the cells, including the nucleus and nucleolus was red, as were also most of the mold filaments and yeast, indicating the presence of considerable artificial coloring matter. The structure indicated that the stock had been manufactured from "trimmings," and further, that they were not fresh when used, but had fermented. There was no oil present. The "new process" is a success in keeping ketchup, as no preservative is mentioned. The price was 20 cents.

No. 112.—Another bottle of the same brand of ketchup; examined in April, 1908; presumably manufactured in 1907; one-twelfth of 1 per cent of sodium benzoate declared on label; a bright red; guaranteed to be from fresh ripe tomatoes and uncolored. The microscope showed no dyeing of the tissues, few fungi, and no extraneous matter. The price was 20 cents.

No. 17.—Opened September 28, 1907; age unknown; a pint bottle; sodium benzoate declared on supplemental label, no amount being stated; reddish brown color, badly discolored on top; greasy odor; not spoiled July 6, 1908; refuse present; large amount of oil; many fungi; the mold filaments enlarged and distorted. The price was 15 cents.

No. 109.—Another bottle of the same brand examined in April, 1908; presumably manufactured the preceding year; had one-tenth of 1 per cent of sodium benzoate; not spoiled July 6, 1908; reddish brown color, discolored near top; greasy odor. This was practically the same as the first bottle examined, had fewer mold filaments, but many bacteria.

No. 18.—Opened September 28, 1907; age unknown; pint bottle; no preservative mentioned; not spoiled July 6, 1908. A neck label stated that it is made from sound

ripe tomatoes and uncolored. Color reddish brown; greasy odor; many oil globules; too many mold filaments and bacteria for sound tomatoes. Price 20 cents.

No. 113.—Another bottle of the same brand examined in April, 1908; said to have been manufactured in 1908; no preservative mentioned; not spoiled after standing open for seventy days; same as No. 18 in color and odor; oil and many fungi again present.

No. 10.—Opened September 2, 1907; age unknown; half-pint bottle; no preservative mentioned; not spoiled July 6, 1908. A neck label 2 inches in height guaranteed the highest quality; an extra label lower down on the neck stated the product to be the natural color, and made from fresh, ripe tomatoes; the regular label carried the brand, manufacturer's name, etc. Color brown; sweetish odor; colonies of mold; distorted filaments; many bacteria; a few small oil globules. Price 25 cents.

No. 106.—Same brand; pint bottle; examined in April, 1908; said to be manufactured in 1907; color red, discolored near surface; 2-inch neck label in addition to regular label; no preservative mentioned; did not spoil in seventy days; oil globules; particles of red, amorphous matter; whole colonies of mold, as well as fragments of filaments; teeming with bacteria.

No. 77.—Different brand, but same manufacturer as Nos. 10 and 106; age unknown; pint bottle; one-twelfth of 1 per cent of sodium benzoate declared; opened December 1; placed in incubator at 95° F. for a month; not spoiled July 6; color reddish brown; greasy odor; oil globules, many mold filaments, and bacteria present. Price 20 cents.

No. 107.—Third brand from same manufacturer as preceding; said to be manufactured in 1907; half-pint bottle; one-twelfth of 1 per cent of benzoate of soda declared; layer of oil on surface; sweet odor; reddish-brown color. Oil globules prominent feature microscopically, whole colonies of distorted mold were present, and sample contained many different forms of bacteria. Price 10 cents.

No. 14.—Opened September 2, 1907; age unknown; no preservative mentioned; not spoiled July 6, 1908; half-pint bottle; color red; good odor; few bacteria; free from refuse. Price 25 cents.

No. 108.—Same brand as No. 14; said to be manufactured in 1907; pint bottle; one-tenth of 1 per cent of benzoate of soda declared; color red; good odor; few fungi; clean and free from refuse.

No. 33.—Opened October 24, 1907; age unknown; one-tenth of 1 per cent of benzoate of soda declared; spoiled November 1; pint bottle (14 ounces); sweetish odor; brown color; many molds, yeast and bacteria. Price 10 cents.

No. 114.—Same brand as No. 33; said to be manufactured in 1907; opened in April; not spoiled in seventy days; many molds, yeasts, and bacteria; some green tissue, and filaments of *algæ*. The price was 10 cents.

SUMMARY.

1. The experiments made during the season of 1907 on the manufacture of tomato ketchup without chemical preservatives were conducted under factory conditions and upon a commercial scale. The results prove that such a ketchup can be made and delivered to the consumer in perfect condition; the product in question having already stood ten months, unopened, without showing the slightest indication of spoilage.

2. The product is of excellent consistency, flavor, and color. The formula employed regularly in the factory where the experiment was

conducted was used, but other recipes could be adapted without changing the character of special brands. In the manufacture of such a product the following precautions were observed:

(a) Whole, sound, ripe tomatoes and high-grade salt, sugar, vinegar, and spices were used; care and cleanliness were observed at every step of the preparation, and the preservation accomplished by heat in the following manner: The pulp was cooked in a steam kettle for about forty minutes, until the mass was reduced to about one-half its volume. Additional processing after bottling did not appear to be necessary to keep the ketchup before opening, and had no effect in these experiments in delaying spoilage after opening.

(b) Ketchup was bottled directly from the cooker at a temperature of 205° F. in bottles prepared in two ways: (1) Sterilized in a steam chamber at 230° F; (2) Washed in hot water, rinsed, and heated to 190° F. in a dry heat for at least thirty minutes. Ketchup was also bottled after the usual process of sieving at 165° F. in bottles prepared in a similar manner. The corks for all bottles were sterilized in a paraffin bath at 270° F. The same ketchup which was bottled at 165° F. was also given subsequent processing at 190° F. and 212° F. for twenty and forty minutes. All have kept without spoilage.

3. Some of the condiments have a limited antiseptic value, but can not be depended upon to prevent spoilage in the quantities used for flavoring. While sugar and vinegar can be added in such amounts as to delay the appearance of molds, and cinnamon and cloves can be depended upon to check deterioration to some extent, these condimental substances have only an incidental value for this purpose.

4. The spoilage of ketchup after opening depends more upon the temperature of the place in which it is kept than on any variation in the manner of processing. Fresh ketchup held, after opening, at a temperature of 95° F. kept for five days on an average without any trace of mold appearing; at 72° it kept for six days; at 67° for eight days; about 46° (refrigerator), fourteen days; and at from 30° to 60° for twenty-seven days. These figures represent the time at which the first trace of spoilage occurred in the neck of the bottle—had this been removed the figures would be much increased—and by no means represent the maximum time during which the ketchup could have been used, the maximum figures, even under these conditions of observation, varying from eight to fifty-eight days. The keeping of the ketchup in warm storage at 70° for one hundred and fifty days before opening hastened the average time of spoilage after opening about one day. The advisability of using small containers, to get the best results with a first-class ketchup, is apparent.

5. Sodium benzoate, even when used in the proportion of 0.1 per cent, is not always effective, and has an injurious effect upon the

living matter of the molds, shown by the distortion and swelling of the filaments, which are filled with a coarse granular protoplasm containing much fat.

6. Artificially colored ketchup can be detected under the microscope by the fact that certain tissues, normally colorless, are dyed red, or by the presence of fine, red, amorphous particles which do not go into solution.

7. Ketchup made from whole ripe stock in a cleanly manner gives a clean appearance under the microscope, but few molds, yeasts, and bacteria being present. On the other hand, ketchup made from trimming stock, or from tomatoes that have been allowed to spoil, contains immense quantities of these growing organisms which may be killed in the process of manufacture, but still give proof of the character of the material used. Ketchup as ordinarily made from trimming stock should, therefore, be designated, so as to differentiate it from that made from sound fresh tomatoes, as the two products are radically different. This exactness in labeling is due no less to the manufacturer than to the consumer, as it is impossible to make the superior product in fair competition with the inferior one, other conditions being equal, unless the two are properly designated, there being naturally some difference in the price.



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Issued March 22, 1909.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY—BULLETIN No. 120.
H. W. WILEY, Chief of Bureau.

THE FEEDING VALUE OF CEREALS, AS CALCULATED FROM CHEMICAL ANALYSES.

By

JOSEPH S. CHAMBERLAIN,
Chief, Cattle-Food and Grain Laboratory, Miscellaneous Division.

IN COLLABORATION WITH THE BUREAU OF PLANT INDUSTRY.



WASHINGTON:
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LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., October 23, 1908.

SIR: I have the honor to transmit for your inspection and approval a manuscript containing the results of a chemical study of the feeding value of certain cereals, especially oats, wheat, and barley, recently completed in the Cattle-Food and Grain Laboratory of this Bureau in collaboration with the Bureau of Plant Industry. I recommend that this report be published as Bulletin No. 120 of the Bureau of Chemistry, its strictly chemical nature suggesting this arrangement, which has the approval of the Chief of the Bureau of Plant Industry.

Respectfully,

H. W. WILEY,
Chief of Bureau.

Hon. JAMES WILSON,
Secretary of Agriculture.

LETTER OF SUBMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY, MISCELLANEOUS DIVISION,
Washington, D. C., October 1, 1908.

SIR: I submit herewith a report on the feeding value of cereals, by Joseph Chamberlain, Chief of the Cattle-Food and Grain Laboratory of the Miscellaneous Division, acknowledgments being due to Mr. H. W. Houghton, assistant chemist, who performed the analytical work on a portion of the samples examined, and to Mr. T. C. Trescot, in charge of the Nitrogen Section of the Bureau of Chemistry, who made the nitrogen determinations. The work was done in collaboration with the Office of Grain Investigations of the Bureau of Plant Industry, having been begun there by the author prior to his transfer to the Bureau of Chemistry. The report discusses the relation between the chemical composition of cereals and their nutritive value, as well as the comparative value of certain varieties of cereals recently introduced.

Respectfully submitted.

J. K. HAYWOOD,
Chief, Miscellaneous Division.

Dr. H. W. WILEY,
*Chief, Bureau of Chemistry,
U. S. Department of Agriculture.*

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THE FEEDING VALUE OF CEREALS AS CALCULATED FROM CHEMICAL ANALYSES.

INTRODUCTION.

The chemical analysis of grains and other materials used in feeding domestic animals consists in the determination of the nutritive substances contained therein. The results of such determinations are expressed in percentage amounts, usually, as pounds per 100 pounds. From these determinations the value of the food for feeding purposes is obtained.

The essential nutritive materials contained in all foods are comprised within the three classes of substances, proteins, fats, and carbohydrates, and the determination of these is the object of a feeding-stuff analysis when its food value is desired. In order that the results of various analyses may be directly comparable the moisture content of the food is found, and from this the amounts of the other constituents are calculated to a uniform water-free, or dry, basis. Another determination is also made, namely, that of the ash or mineral matter. The mineral substances present in a food play an important part in the physiological process of digestion, but possess no value as a direct source of energy. Their action is mainly indirect, and as no method of measuring their value is known, they are not considered in calculating the nutritive value of foods. The usual form, therefore, of expressing the results of an analysis of a stock food is to give the amounts of the following substances present: Water, ash, protein, fat, and carbohydrate; the food value of the material being based on the last three.

In the study of such complex materials as feeding stuffs, however, it has been found that a knowledge of the total amounts of these three nutritive constituents is not sufficient to give an exact idea of the food value of any substance. This is due to factors depending, first, on the character of the food itself and, second, on that of the animal using it. The three groups of substances—proteins, fats, and carbohydrates—do not yield all of their material to the animal as nutriment, nor are they nutritive in equal degrees. The proportion that is actually digested and used by the animal is different in each case, i. e., each possesses a certain coefficient of digestibility. Furthermore, each of these groups as ordinarily determined contains separate and distinctly different substances which, under certain conditions or when present in small amounts, may be disregarded, but which, when present in large amounts or when productive of decided effects, even though in small quantities, must be considered separately.

NUTRIENTS ON WHICH THE FEEDING VALUE IS BASED.**PROTEINS.**

This class of substances includes the true proteins, which are of an organic nitrogenous nature, very complex and similar in composition and properties to white of egg or lean meat. They are of very great importance, for from them the animal derives the nitrogenous material from which its own muscular tissue is built. The separation of the proteins into individuals or even into classes is very difficult and, though they are not all equal in nutritive value, the investigations throwing light upon this question are so recent and incomplete that it is impossible, at present, to attempt any separation. With vegetable foods the proteins may be left as a group, because those present are the simple proteins and are similar to each other.

There is, however, another class of nitrogenous organic bodies which are not proteins, but which are included with them in determinations made by the usual methods. These substances are the amido compounds. Many of the proteins contain approximately 16 per cent of nitrogen, and it is customary in analyzing protein foods to determine simply the amount of nitrogen present. The result obtained by multiplying this amount by 6.25 is called crude protein. When determined in this way, crude protein includes the amido compounds, which do not contain as much nitrogen as the proteins, nor do they furnish as much, if any, nutriment to the animal. In cereal grains the amount of these amido compounds present is very small, even within the limits of error of the method of separation used,^a and on this account they are disregarded and the crude protein is considered as true protein.^b

FATS.

The method for the determination of fats is that of extraction with ether, and all substances soluble in ether, such as chlorophyll, organic acids, waxes, resins, etc., are included. In some materials allowance would have to be made for such a mixture, but in grains and a great number of ordinary stock foods it is not necessary. The extracted product is not always a pure fat or a mixture of pure fats, but when waxes or resins are present they are in small quantities and their nutritive value is probably almost the same as that of true fats. No great error is introduced, therefore, if the extract is considered as fat, and this is usually done. On account of the impure nature of the extract, the name "ether extract" is often used instead of the term

^a Stutzer's method.

^b The protein factor used in this investigation, except in the case of wheat, is the common one, viz, 6.25. With wheat the now accepted factor of 5.7 has been adopted.

"fat." In this bulletin, however, the term "fat" is used to signify the crude product obtained by the ordinary extraction of the dry material with anhydrous ether.

CARBOHYDRATES.

In an ordinary analysis of stock foods or similar material all those substances left after the ash, protein, and fat have been determined are included in the term "nitrogen-free extract" and considered as carbohydrate. In materials containing much sugar or when it is desirable to know the amount of starch, as such, these constituents are determined separately. In cereal grains, excepting sweet corn or saccharine sorghums, the amount of sugar is not great and starch is practically the only carbohydrate present. In such cases no separate determination of these substances is made and the total carbohydrate is determined only by difference, i. e., 100 per cent less the amount of ash, protein, and fat combined.

In many vegetable products, however, a considerable portion of this nitrogen-free extract or carbohydrate remainder consists of fibrous material, such as hulls, stalks, and the cellulose portions of plants generally. This material, though a true carbohydrate in nature, has a distinctly different coefficient of digestibility as compared with sugar or starch, and must therefore be considered separately. This coarse fibrous portion of vegetable foods is termed "crude fiber," and though only slightly digestible it plays a very important part in digestion. It acts as a dilutant of the more concentrated portions of the food, such as starch, necessitates for the whole food thorough mastication, and prevents it from becoming too compact; in other words, it keeps the food mass porous and open to the action of the digestive fluids. If, however, the amount of this crude fiber or roughage is in excess, as in the case of coarse fodders, the amount of energy expended by the animal in securing and digesting the food is so great that its ultimate nutritive value is correspondingly diminished. That part of the crude fiber which is digested has, however, the same nutritive value as the other carbohydrates, so that, except as regards digestibility, the crude fiber is considered with the other carbohydrates.

Another carbohydrate substance, in addition to sugar, starch, and crude fiber, is sometimes considered separately, namely, the pentosans. These are compounds related to the sugars, being polymeric forms of five carbon sugars. Their digestibility and nutritive value, according to Kellner,^a are nearly the same as those of starch, and consequently a separate determination is not necessary. When sugar is present in any considerable amount it must be determined separately, as its nutritive value is slightly different from the other carbohydrates.

^a Kellner, *Die Ernährung der landwirtschaftlichen Nutztiere*, 1905, pp. 88, 153.

The remaining substances sometimes present and included in the nitrogen-free extract, namely, the organic acids, are seldom found in sufficient amount, especially in ordinary fresh foods, to make their consideration necessary.

CLASSIFICATION OF ALL SEPARATIONS.

On account, therefore, of differences in digestibility or in nutritive value, the analytical separation of a feeding material as previously given must be enlarged to include part or all of the following substances:

Proteins:

True proteins.

Amido compounds.

Fats:

True fats.

Waxes, resins, organic acids, chlorophyl, etc.

Carbohydrates:

Sugars, starch, pentosans, and organic acids.

Crude fiber.

In the case of cereal grains and similar materials this classification for separation may be condensed, because of the small amounts of most of the secondary constituents which are present, as follows: Protein, fat, crude fiber, carbohydrate. In the present investigation these are the determinations that have been made.

FACTORS USED IN CALCULATING FOOD VALUES.

COEFFICIENT OF DIGESTIBILITY.

The separation of the nutritive constituents of foods into the groups or classes just described is made because of the differences in their relative digestibility. The effect of the general nature of the food on its digestibility must also be considered. Each of the nutritive constituents, protein, fat, crude fiber, and carbohydrate, has a coefficient of digestibility which differs for each food in which it occurs. Therefore the coefficient of digestibility of each of these constituents must be known for each food under consideration. Such coefficients have been determined by actual feeding experiments on living animals and factors of digestibility calculated from the results. It has also been shown that it is necessary to consider the animal itself as well as the food and the individual constituents. Such experimental results can not be obtained in all cases, but they have been secured by a few investigators for the more common domestic animals and for a considerable number of foods. From the results of such direct experiments the general factors thus obtained may be applied to a variety of foods. The results obtained by Kellner at the Möckern experiment station are considered reliable and

have been used in the present investigation. The coefficients of digestibility as given in his tables ^a may be illustrated as follows:

When oats are fed to horses the following amounts are digested:		Per cent.
Protein.....	80	
Fat.....	71	
Crude fiber.....	29	
Carbohydrate.....	75	
When fed to mature fattening cattle the amounts are:		
Protein.....	78	
Fat.....	83	
Crude fiber.....	26	
Carbohydrate.....	77	

If these coefficients are applied to a particular case—for example, a sample of oats—the following percentages of the nutrients are found to be available under the conditions specified:

TABLE I.—*Digestibility of oats for horses and cattle.*

Nutrient.	Total.	Digestible.	
		For horses.	For mature fattening cattle.
	Per cent.	Per cent.	Per cent.
Protein.....	12.80	10.32	10.00
Fat.....	4.15	2.95	3.44
Crude fiber.....	12.53	3.63	3.25
Carbohydrate.....	66.58	49.94	51.27

These figures express the mean results of actual feeding experiments and, while variations will necessarily be introduced by each individual animal, they may still be considered as giving fair average results applicable in ordinary cases.

The following tables give the coefficients of digestibility used in this bulletin, the amounts of crude nutrients as found by analysis, and the digestible nutrients as calculated from these:

TABLE II.—*Coefficients of digestibility.*

[For mature fattening cattle.]

Grain.	Protein.	Fat.	Crude fiber.	Carbohydrate.
	Per cent.	Per cent.	Per cent.	Per cent.
Oats ^b	78	83	26	77
Emmer ^c	75	71	44	75
Einkorn ^c	75	71	44	75
Wheat ^d	84	63	47	92
Rye ^b	84	64	53	92
Barley ^b	70	80	33	92
Proso ^e	49.2	76.9	68.3	85.2
Sorghum ^f	65	70	51	91
Maize ^b	72	90	41	95

^a Kellner, Die Ernährung der landwirtschaftlichen Nutztiere, 1905, pp. 561, 569.

^b Ibid., p. 569.

^c Ibid., p. 561, "Spelzweizen."

^d Ibid., p. 561.

^e Tangl, Landw. Jahrb., 1905, 34:1, "Besenhirsekorn" fed to oxen.

^f Kellner, *ibid.*, p. 569, "Darl."

TABLE III.—*Total and digestible nutrients.*

[Pounds per hundred pounds.]

Grain.	Protein.		Fat.		Crude fiber.		Carbohydrate.	
	Total.	Digestible.	Total.	Digestible.	Total.	Digestible.	Total.	Digestible.
Oats.....	13.76	10.73	4.33	3.59	12.20	3.17	66.39	51.04
Emmer.....	13.28	9.86	1.91	1.36	11.31	4.98	69.42	52.06
Einkorn.....	14.07	11.00	2.19	1.55	13.55	5.96	64.02	48.04
Wheat.....	14.20	11.93	2.36	1.49	2.78	1.30	78.72	72.42
Rye.....	13.44	11.29	1.83	1.17	2.30	1.22	80.24	73.82
Barley.....	13.39	9.37	1.87	1.66	5.64	1.86	76.05	69.86
Proso.....	12.77	6.28	3.27	2.51	8.95	6.11	71.23	60.69
Sorghum.....	11.71	7.61	3.25	2.27	1.80	.92	81.58	74.24
Maize.....	9.91	7.14	4.40	3.92	2.21	1.28	81.96	77.86

CALORIFIC VALUE AND METABOLIZABLE ENERGY.

When the nutrients of food are utilized within the animal body, the ultimate product is either flesh or energy. The unit for measuring this flesh or energy-producing power of a food is the unit of heat energy or the calorie and the value so obtained is termed its calorific or fuel value.

The actual measurement of this calorific value is made in an instrument called a calorimeter, in which an exact amount of substance (e. g., one of the nutrient constituents of foods, such as fat) is burned in an atmosphere of oxygen and the amount of heat so generated is measured and expressed in calories.

When 1 gram of water is raised 1° C. in temperature, the amount of heat used is called a calorie. As this unit is too small for most purposes, it is multiplied by 1,000; that is, the amount of heat required to raise the temperature of 1,000 grams or 1 kilogram of water 1° C. In English units this is equivalent to the amount of heat required to raise the temperature of 1 pound of water 4° F. This unit is called the large Calorie, and is written capitalized to distinguish it from the small calorie.

The total calorific value of a food is equal to the sum of the calorific values of the different nutritive constituents. The entire food is, however, not utilized by the animal, and the calorific value of each nutrient must be determined not on the total amount of nutrient present, but only that part which is digestible. Also, in the processes of digestion there are formed certain intermediate waste products that still possess potential energy, and the energy of the digestible portion of the food must be corrected by the amount of energy in these waste products in order to determine the true amount of energy in the food actually utilized by the animal. This resultant energy

utilized by the animal has been termed available energy or, better, metabolizable energy.^a

The loss of energy due to waste products other than undigested food varies with different animals and with each of the nutrients, so that the corrections necessary can only be made after direct experiment. Such experiments have been made upon carnivorous animals (dogs) by Rübner, and his results giving the available or metabolizable energy of each of the three groups of nutrients have been accepted and applied to other animals as well. Within recent years, however, Kellner and others have carried out experiments with other animals, especially herbivorous animals, and it has been found that the figures given by Rübner for dogs do not apply to herbivorous animals. Kellner's work was with mature fattening cattle, and the following table gives the total calorific value and metabolizable energy in Calories per pound for each of the three groups of nutrients as found by Kellner and the metabolizable energy as found by Rübner. In Kellner's work the corrections were made on the values for wheat gluten as protein, on ordinary ether extract, and on starch as carbohydrates.

TABLE IV.—*Metabolizable energy of nutrients per pound.*

Nutrient	Cattle (Kellner).		Dogs (Rübner)
	Total calorific value.	Metabolizable energy.	Metabolizable energy.
	<i>Calories.</i>	<i>Calories.</i>	<i>Calories.</i>
Protein.....	2,720	2,220	1,860
Ether extract.....	3,780	3,780	4,220
Carbohydrates.....	1,900	1,710	1,860

These figures mean that for every pound of protein digested by cattle 2,720 Calories of energy are produced, but only 2,220 Calories are really available from the food metabolized by the animal for the production of energy or flesh. From the ether extract 3,780 Calories of energy are produced for every pound digested, and of this the entire amount represents metabolized food. The carbohydrates possess 1,900 Calories per pound of total energy, while only 1,710 Calories are yielded by the food really metabolized.

From Table III (p. 10), which gives the amount of total and digestible nutrients, the energy yielded by the metabolized food for the production of animal energy or flesh can be calculated. The following table gives the total nutrients, digestible nutrients, and

^a Armsby, Principles of Animal Nutrition, 1906, p. 270.

metabolizable energy. The last is calculated both according to the figures of Kellner and of Rübner, for the purpose of comparison:

TABLE V.—*Metabolizable energy.*

[Pounds per hundred pounds.]

Grain.	Protein.				Fat.			
	Total nutrients.	Digestible nutrients.	Fuel value.		Total nutrients.	Digestible nutrients.	Fuel value.	
			Calories (Kellner).	Calories (Rübner).			Calories (Kellner).	Calories (Rübner).
Oats.....	13.76	10.73	19,958	23,820	4.33	3.59	15,150	13,570
Emmer.....	13.28	9.96	18,525	22,110	1.91	1.36	5,740	5,141
Einkorn.....	14.67	11.00	20,460	24,420	2.19	1.55	6,542	5,860
Wheat.....	14.20	11.93	22,190	26,490	2.36	1.49	6,288	5,632
Rye.....	13.44	11.29	21,000	25,080	1.83	1.17	4,938	4,423
Barley.....	13.39	9.37	17,428	20,800	1.89	1.66	7,005	6,275
Proso.....	12.77	6.28	11,680	13,940	3.27	2.51	10,592	9,488
Sorghum.....	11.71	7.61	14,134	16,890	3.25	2.27	9,580	8,581
Maize.....	9.91	7.14	13,280	15,850	4.40	3.92	16,542	14,820

Grain.	Crude fiber.				Carbohydrate.				Total fuel value.	
	Total nutrients.	Digestible nutrients.	Fuel value.		Total nutrients.	Digestible nutrients.	Fuel value.		Calories (Kellner).	Calories (Rübner).
			Calories (Kellner).	Calories (Rübner).			Calories (Kellner).	Calories (Rübner).		
Oats.....	12.20	3.17	5,896	5,421	66.29	51.04	94,940	87,280	135,944	130,091
Emmer.....	11.31	4.98	9,263	8,516	69.42	52.06	96,830	89,020	140,358	124,787
Einkorn.....	13.55	5.96	11,065	10,190	64.02	48.04	89,320	82,150	127,407	122,620
Wheat.....	2.78	1.30	2,418	2,223	78.72	72.42	134,690	123,840	165,586	158,185
Rye.....	2.30	1.22	2,269	2,086	80.24	73.82	137,320	126,230	165,527	157,799
Barley.....	5.64	1.86	3,460	3,181	76.05	69.96	130,120	119,630	158,013	149,888
Proso.....	8.95	6.11	11,364	10,450	71.23	60.69	112,880	103,780	146,516	137,658
Sorghum.....	1.90	0.92	1,711	1,573	81.58	74.24	138,080	126,950	163,525	153,994
Maize.....	2.21	1.28	2,380	2,190	81.96	77.86	144,820	133,140	177,022	166,000

As before stated, the metabolizable energy of a food is the total amount of energy which an animal is able to derive from that portion of food that is digested and actually used by it after allowing for all waste. This was originally termed the available energy of the food and was considered to be a measure of the value of the food. There are, however, still other factors entering into the problem that need to be considered which make it necessary to distinguish between this available or metabolizable energy and the net available energy of the food.

The animal body has been likened by Armsby to a steam boiler fed by means of a mechanical stoker, the power for operating which comes from the steam generated in the boiler itself. The available energy of the coal for producing steam power is equivalent to the fuel value of the coal less that of the refuse and again diminished by the amount of energy used in operating the mechanical stoker. Similar conditions obtain in the animal body. In order to get the food and chew and digest it, a certain amount of the animal energy

must be used, and the real available energy of the food is therefore equivalent to the metabolizable energy, or gross available energy, less the energy expended during the eating and digestion.

PRODUCTION VALUE.

The finally available energy of the food may be of value in two ways, depending on the object for which the animal is fed. It may be desired simply to prevent the loss of flesh or energy, i. e., the object may be to maintain the animal in all respects in a certain desirable condition. Food given for such a purpose is known as a "maintenance ration," and each of the nutrients has a value as a part of such a ration, which value can only be determined by experiments on animals, for it has been found that the metabolizable energy and the energy as maintenance are not equal. This work has been done by Armsby,^a who found in general that 62.92 per cent of the metabolizable energy of clover hay was available for maintenance.

On the other hand, a ration may be fed to animals for the purpose of producing increase in flesh, and consequently more than a maintenance ration must be given. In this case, that part of the food that is in excess of what is required for maintenance is used by the animal for increase in flesh and has a definite value as such. In experiments to determine the value of a given food for producing flesh, the material is fed in addition to a basal maintenance ration and its value determined as measured by the gain in flesh produced. The value so found has been termed the *production value* of the food. Kellner^b has determined these production values for the pure nutrients when fed in addition to a basal maintenance ration to mature fattening cattle. He found that while the ratio of the nutrients to each other remained nearly the same the actual values for production are only about one-half the metabolizable values.

TABLE VI.—Comparison of metabolizable energy and production value of nutrients per pound.

Nutrient.	Metabolizable energy.	Production value.
	Calories.	Calories.
Protein.....	2,220	1,016
Ether extract.....	3,780	2,273
Carbohydrates.....	1,710	1,071

The most direct and the simplest method of expressing this production value is in Calories per pound or per 100 pounds, as is done by Armsby^c in recent publications of the Pennsylvania agricultural

^a Pennsylvania Agr. Exp. Sta. (1905), Bul. 71, p. 12.

^b Loc. cit., p. 153.

^c Loc. cit.

experiment station in cooperation with the Bureau of Animal Industry of the Department of Agriculture. In Kellner's work, however, the values are expressed in different terms. It has been found^a that the production values in Calories for the various nutrients correspond to the production of a definite weight of body tissue, chiefly fat. The figures so obtained are as follows:

TABLE VII.—*Production value of nutrients of foods.*

Nutrient.	Production value.	
	Calories per pound.	Pounds flesh gained per 100 pounds nutrient.
Protein.....	1,016	23.5
Ether extract:		
Coarse fodders.....	2,041	47.4
Grains.....	2,273	52.6
Concentrated foods.....	2,585	59.8
Starch and crude fiber.....	1,071	24.8
Sugar.....	812	18.8

If one of these nutrients be selected as a standard and its value, stated in Calories or in pounds flesh gained, be regarded as unity, the total production value may also be expressed in terms of this unit. Starch is the nutrient that has been taken as unity in this case, and its value of 1,071 Calories per pound, or 24.8 pounds of fat gained per hundred pounds, is the unit of measurement. The value of each nutrient in pounds of starch is calculated, and from this the total value of the food in pounds of starch, equivalent to 100 pounds of the food, is obtained. This is termed the starch value (or "Stärkewert").

There are, then, three terms in which the production value of a food may be expressed: (1) Calories per 100 pounds; (2) pounds of flesh gained per 100 pounds; (3) starch value per 100 pounds. These three methods of expression are illustrated and shown comparatively in Table VIII, giving the digestible nutrients and the individual and total production values in terms of each unit described.

TABLE VIII.—*Production value of various foods expressed according to three methods.*

[Pounds per hundred pounds of dry matter.]

Grain.	Protein.				Ether extract.			
	Digestible.	Production value.			Digestible.	Production value.		
		Calories.	Flesh gained.	Starch value.		Calories.	Flesh gained.	Starch value.
Oats.....	10.73	10,893	2.52	10.2	3.59	8,171	1.89	7.6
Emmer.....	9.96	10,127	2.34	9.4	1.36	3,068	.71	2.9
Einkorn.....	11.00	11,153	2.58	10.4	1.55	3,502	.81	3.3
Wheat.....	11.93	12,233	2.83	11.4	1.49	3,372	.78	3.1
Rye.....	11.29	11,457	2.65	10.7	1.17	2,637	.61	2.5
Barley.....	9.37	9,511	2.20	8.9	1.66	3,761	.87	3.5
Proso.....	6.28	6,398	1.48	6.0	2.51	5,706	1.32	5.3
Sorghum.....	7.61	7,738	1.79	7.2	2.27	5,144	1.19	4.8
Maize.....	7.14	7,263	1.68	6.8	3.92	8,906	2.06	8.3

^a Kellner, loc. cit., pp. 153, 379.

TABLE VIII.—*Production value of various foods expressed according to three methods—Con.*

Grain.	Crude fiber.				Carbohydrates.				Total production value.		
	Digestible.	Production value.			Digestible.	Production value.					
		Calo-ries.	Flesh gained.	Starch value.		Calo-ries.	Flesh gained.	Starch value.	Calo-ries.	Flesh gained.	Starch value.
Oats.....	3.17	3,415	0.79	3.2	51.04	54,730	12.66	51.0	77,209	17.86	72.0
Emmer.....	4.98	5,360	1.24	5.0	52.06	55,810	12.91	52.1	74,356	17.20	69.4
Einkorn.....	5.96	6,398	1.48	6.0	48.04	51,620	11.94	48.1	72,673	16.81	67.8
Wheat.....	1.30	1,383	.32	1.3	72.42	77,642	17.96	72.4	94,500	21.86	88.1
Rye.....	1.22	1,297	.30	1.2	73.82	71,958	18.31	73.8	94,544	21.87	88.2
Barley.....	1.86	1,969	.46	1.8	69.96	75,005	17.35	70.0	90,264	20.88	84.2
Proso.....	6.11	6,571	1.52	6.1	60.69	65,062	15.05	60.7	83,736	19.37	78.1
Sorghum.....	.92	9,943	.23	.9	74.24	79,590	18.41	74.2	93,463	21.62	87.2
Maize.....	1.28	1,383	.32	1.3	77.86	83,480	19.31	77.9	100,985	23.36	94.2

The advantage in expressing the production values in Calories per hundred pounds is that the results are easily comparable, whether the object of feeding is the production of flesh, as with fattening cattle, or the production of muscular energy, as with work horses. The disadvantage is that the unit is so small that the number expressing the final value is too large to convey a distinct idea concerning it. Numbers of five and six figures are difficult to grasp readily, and though differences are at the same time magnified the real meaning of the results is harder to comprehend with large numbers than with smaller ones. It is also true that to the average mind not trained in scientific thought the term "Calories per hundred pounds" is much more vague than either "pounds of flesh" gained or even the "starch value." The last, while expressed in numbers which are as easily compared as those expressing pounds of flesh gained, is not quite so commonly understood and, though readily transferable for either flesh or energy production, it has been set aside, the preference being given to the expression "pounds of flesh gained per hundred pounds of food." This term is readily understood by all who feed stock and is definite and applicable in the majority of cases.^a

On account of these facts, the production values for each nutrient and for the whole food have been calculated and expressed in this investigation in terms of "pounds of flesh gained per hundred pounds of food." Attention must be called to the fact that this term does not mean exactly pounds of live weight gained, for the latter includes with the gain in the flesh or fat the increase in water also and will therefore always be somewhat higher. However, while not exactly synonymous, the two terms are approximately the same and express the increase in flesh or weight as the result of feeding 100 pounds of food in addition to a basal maintenance ration.

^aSince the above was written, Armsby (Science, 1907, 26: 670) has proposed a new unit for the energy value of rations, namely, the "Therm=T," equivalent to 1,000 large Calories, or 1,000,000 gram calories.

COMPARATIVE NUTRITIVE VALUE AND NUTRITIVE RATIO.

According to the figures given by Rübner for the metabolizable energy of the nutrients of food, protein and starch have an equal value, namely, 1,860 Calories, while the ether extract has 2.25 times as much, namely, 4,220 Calories. This relation has led to the use of two factors for valuation based directly on the amount of digestible nutrients present. If the amount of ether extract is multiplied by 2.25 it is then measured on the same scale as the other nutrients and the sum of the amounts of all the nutrients is therefore directly comparable, one food with another. The total amount of nutrients so found is known as the comparative nutritive value; that is, comparative nutritive value = protein + carbohydrate + (ether extract $\times$ 2.25). The figures thus obtained express the relative value of foods measured by the amount of digestible nutrients present.

Another factor based on these facts is the nutritive ratio which expresses the relative amounts, in a food, of the two classes of nutrients, namely, the heat-producing nutrients and those producing flesh or tissue. The proteins of a food are the constituents which build up the tissue of the animal and are productive of muscular energy. The carbohydrates and fats, on the other hand, are chiefly heat or fat producers. The ratio between these two groups has been long considered as of value in indicating the general character of the food as fattening and heat producing or as productive of increased muscular tissue and energy.

This ratio, "protein : (carbohydrates + ether extract $\times$ 2.25) :: 1 : x," is termed "broad" when it is :: 1 : 8-12 as in maize and indicates a predominance of heating or fattening elements; and it is termed "narrow" when it is :: 1 : 4-7, as in oats, in which the constituents producing muscular tissue are in excess. The comparative nutritive value and the nutritive ratio of these grains, which have been discussed in the preceding tables, are shown in Table IX.

TABLE IX.—Comparative nutritive value and nutritive ratio of grains.

Grain.	Comparative nutritive value.	Nutritive ratio.	Grain.	Comparative nutritive value.	Nutritive ratio.
Oats.....	73.01	5.8	Barley.....	84.92	8.0
Emmer.....	70.06	6.0	Proso.....	78.73	11.5
Einkorn.....	68.49	5.2	Sorghum.....	87.88	10.5
Wheat.....	89.00	6.5	Maize.....	95.10	12.3
Rye.....	88.96	6.9			

Table IX shows clearly the significance of the term "nutritive ratio" as exemplified by the two characteristically different grains maize and oats, the former being a heat-producing food with a ratio 1 : 12.3, and the latter a tissue-forming food whose ratio is 1 : 5.8.

PLAN OF THE PRESENT INVESTIGATION.**METHOD OF ESTIMATING THE RELATIVE FOOD VALUES OF GRAINS.**

While the direct experimental method is without question the one on which to rely for the determination of the maintenance or production values of feeding stuffs, it is not always possible to make such investigations on living animals with a large number of foods. Such direct experiments have been made both in this country and Europe by the investigators previously mentioned, and their work embraces quite a number of different animals and foods. Taking the results of these investigations as a basis, and using the factors that have thus been found to apply to certain animals and certain foods, it is possible to calculate from simple analytical data the production values of a larger number of samples of feeding stuffs than could possibly be determined in any other way. The production values and nutritive ratios of foods thus determined form the best basis now available for a comparative or even for an actual valuation of stock foods.

In the present investigation of the feeding value of cereal grains this method has been used to determine the relative value of the different grains studied. It is not claimed that these results are as accurate as those that would have been obtained had careful feeding experiments been made with each sample, but as the factors used in calculating the final values are based upon average results with each group or class of grain studied, the calculated values are the best obtainable from simple analytical results. Neither total fuel value, total metabolizable energy, nor comparative nutritive value have been calculated, because with grains, where the chief object in feeding is the production of flesh, the production value and nutritive ratio express the values best. As stated previously, the factors used are those given by Kellner, except in the case of sorghum, to which material the recent results of Tangl are more applicable. For coefficients of digestibility as given in Table II (p. 9), figures for the identical species of cereal were used when this was known without question, as with oats, barley, rye, wheat, and maize. For emmer and einkorn the figures are those for the German "Spelzweizen," which is identical with one, but not with the other. The two grains are, however, so similar that little error is probably introduced. For nonsaccharine sorghums, the figures are those for "Dari," and for proso, Tangl's figures for "Besenhirsekorn," fed to oxen. For production values the factors of Kellner given in Tables VI and VII (pp. 13 and 14) are used.

DESCRIPTION OF MATERIAL EXAMINED.

The material examined in the present investigation consists of samples of different grains that have been under field trial by the Office of Grain Investigations of the Bureau of Plant Industry dur-

ing the last few years. The following cereal grains are represented: Oats, emmer, einkorn, wheat, rye, barley, proso, sorghum, and maize. Under each class the samples consist, when possible, of both the original imported seed and grain grown from this seed in various sections of the United States.

The greater part of the work on the introduction of new cereal grains has had to do with the semiarid regions of this country, the cereals introduced having been selected either on account of (1) drought-resistant qualities, or (2) rust or disease resistance, in addition to their yield and general character. Whether or not these qualities are maintained on introduction into this country can only be determined by field tests, moreover, two questions arise in regard to these introduced grains: (1) Are they of equal or superior quality to domestic varieties, and (2) do they maintain their characteristic properties and qualities when grown for successive years in the United States.

The analyses made in this investigation and their correlation for the relative valuation of the samples studied are an attempt to answer these questions without entering into a study of the direct effect of change of environment on the properties of cereals.

DISCUSSION OF RESULTS.

The complete tabular results of all analyses with the calculated production values for each nutrient and for the whole food are given in the tables at the close of the bulletin (p. 48). The discussion of tables of average results are presented in the following pages, each cereal being discussed under a separate caption.

OATS (*Avena sativa*).

COMPARISON OF DOMESTIC AND FOREIGN OATS.

The present investigation was undertaken primarily to study the properties and composition of the Swedish Select oat, a very desirable variety, the seed of which was introduced from Russia in 1899 by the Office of Grain Investigations, U. S. Department of Agriculture. This oat is especially resistant to drought and rust, is strong and hardy, and makes a good yield. It answers, therefore, particularly well all the requirements for introduction.

Among the oats analyzed there are 128 samples of the Swedish Select oat grown in various sections of the United States. In addition to these, there are 89 samples from 13 varieties of introduced oats which have been grown in the United States for a short time, usually not more than three years. There are also 25 samples, representing 10 varieties of purely domestic oats. This makes 242 samples, representing 24 varieties of oats grown in the United States either from purely domestic seed or from recently introduced

foreign seed. For comparison with these analyses, 54 samples, representing 37 varieties of strictly foreign-grown oats, were analyzed, including most of the varieties represented in the two classes of introduced oats just mentioned.

This selection of samples makes it possible to compare domestic and foreign grown oats in general, as well as samples of the same variety grown in Europe and in this country and different varieties of introduced grain that have been grown in the United States for several years.

The composition of a typical domestic oat, expressed both as total and digestible nutrients, has been given as follows:^a

TABLE X.—*Typical United States oats.*

[Recalculated to dry basis per hundred pounds.]

Nutrient.	Total.	Digestible.
	<i>Pounds.</i>	<i>Pounds.</i>
Moisture.....	10.00	
Protein.....	13.33	10.40
Fat.....	5.00	4.15
Crude fiber.....	13.33	3.46
Carbohydrate.....	64.44	49.62

The mean of 72 samples of United States and Canadian oats analyzed at the Columbian Exposition in Chicago in 1893 is given in Table XI.^b

TABLE XI.—*United States and Canadian oats, 1893.*

[Recalculated to dry basis per hundred pounds.]

Nutrient.	Total.	Digestible.
	<i>Pounds.</i>	<i>Pounds.</i>
Moisture.....	9.96	
Protein.....	13.40	10.45
Fat.....	4.91	4.07
Crude fiber.....	13.24	3.44
Carbohydrate.....	64.73	49.84

The mean of 25 samples of purely domestic oats studied in this investigation has been found to be as follows:

TABLE XII.—*United States oats, 1906.*

[Per hundred pounds of dry matter.]

Nutrient.	Total.	Digestible.
	<i>Pounds.</i>	<i>Pounds.</i>
Moisture.....	8.20	
Protein.....	13.40	10.60
Fat.....	5.62	4.67
Crude fiber.....	11.46	2.98
Carbohydrate.....	65.18	50.19

^a U. S. Dept. Agr., Bureau of Chemistry, 1898, Bul. 13, Pt. IX, p. 1180.^b U. S. Dept. Agr., Bureau of Chemistry, 1895, Bul. 45, p. 29.

The 128 samples of Swedish Select oats grown in the United States were found to have a mean composition as follows:

TABLE XIII.—*Swedish Select oats.*

[Per hundred pounds of dry matter.]

Nutrient.	Total.	Digestible.
	<i>Pounds.</i>	<i>Pounds.</i>
Moisture.....	7.10	
Protein.....	12.85	10.02
Fat.....	4.00	3.32
Crude fiber.....	12.23	3.18
Carbohydrate.....	66.99	51.58

The mean of 242 samples of recently introduced miscellaneous varieties grown in the United States was found to be as follows:

TABLE XIV.—*Miscellaneous introduced varieties of oats.*

[Per hundred pounds of dry matter.]

Nutrient.	Total.	Digestible.
	<i>Pounds.</i>	<i>Pounds.</i>
Moisture.....	7.82	
Protein.....	13.76	10.73
Fat.....	4.33	3.59
Crude fiber.....	12.20	3.17
Carbohydrate.....	66.29	51.04

Fifty-four samples of foreign-grown oats, including most of the varieties represented in Table XIV, were found to have the mean composition shown in Table XV.

TABLE XV.—*Foreign oats.*

[Per hundred pounds of dry matter.]

Nutrient.	Total.	Digestible.
	<i>Pounds.</i>	<i>Pounds.</i>
Moisture.....	8.31	
Protein.....	11.63	9.07
Fat.....	5.86	4.86
Crude fiber.....	10.89	2.83
Carbohydrate.....	67.91	52.28

Table XVI gives the digestible nutrients, the total production values, and the nutritive ratios of these representative groups, together with a few analyses obtained from other sources, the last named being calculated from the original analyses reported in the places cited.

TABLE XVI.—Group values for digestible nutrients, production value, and nutritive ratio of oats, domestic and foreign.

[Pounds per hundred pounds of dry matter.]

Description of grain.	Number of samples.	Digestible nutrients.				Production value.		Nutritive ratio.
		Protein.	Fat.	Crude fiber.	Carbohydrate.	Calories.	Gain in flesh.	
Typical United States, 1898 ^a		10.40	4.15	3.46	40.62	77,395	17.88	6.0
Mean, United States and Canada, 1893 ^b	72	10.45	4.07	3.44	40.84	77,049	17.80	6.0
Miscellaneous, United States, 1906	242	10.73	3.59	3.17	51.04	77,209	17.86	5.8
Strictly United States, 1906.	25	10.60	4.67	2.98	50.19	78,377	18.11	6.0
Swedish Select, United States, 1906	128	10.02	3.32	3.18	51.58	76,431	17.68	6.2
Miscellaneous European, 1906	54	9.07	4.86	2.83	52.28	79,327	18.35	7.3
Foreign ^c		9.26	4.59	3.09	51.09	78,510	18.17	7.0
Foreign ^d	307	9.07	5.02	2.93	51.57	79,024	18.28	7.2

^a U. S. Department of Agriculture, Bureau of Chemistry, 1898, Bul. 13, Pt. IX, p. 1180.^b U. S. Department of Agriculture, Bureau of Chemistry, 1895, Bul. 45, p. 29.^c Kellner, loc. cit., p. 561, recalculated to dry basis.^d König, *Chemie der menschlichen Nahrungs- und Genussmittel*, 1903, p. 533, recalculated to dry basis.

It will be seen from these data that there is a distinct difference between the foreign-grown oats and those grown in the United States. The foreign-grown oats, such as the miscellaneous samples reported by König, the standard used by Kellner, and the 54 samples of foreign-grown oats analyzed in the present investigation, show these differences plainly. This variation in composition may be stated as follows: Oats grown in the United States are characterized, as a rule, by higher protein, lower carbohydrate, lower fat, and higher crude fiber. These differences in composition affect the production value and the nutritive ratio, often making the former lower in the oats grown in the United States and the latter narrower. The differences in the production values are not great, however; the maximum difference in pounds of flesh gained between the domestic-grown Swedish Select oat (17.68 pounds) and the miscellaneous foreign oats analyzed in this bulletin (18.35 pounds) being only 0.67 pound, while the minimum difference between the highest domestic oat and the lowest foreign oat was only 0.06 pound of flesh gained per hundred pounds of oats fed. The results obtained in the present study afford a direct comparison of the foreign oat and the same oat grown in the United States, since in most cases the domestic-grown oat was obtained from the same seed as the sample from which the analysis of the foreign oat was made.

TABLE XVII.—*Comparison of domestic and foreign grown oats of the same varieties.*

[Per hundred pounds of dry matter.]

Variety.	Number of samples.	Digestible nutrients.				Production value.		Nutritive ratio.
		Protein.	Fat.	Crude fiber.	Carbohydrae.	Calories.	Gain in flesh.	
Swedish Select:		<i>Lbs.</i>	<i>Lbs.</i>	<i>Lbs.</i>	<i>Lbs.</i>		<i>Lbs.</i>	
Foreign.....	3	9.89	3.62	2.98	52.89	78,160	18.08	6.5
United States.....	128	10.02	3.32	3.18	51.58	76,431	17.68	6.2
Belgian Winter:								
Foreign.....	4	8.76	5.66	2.28	54.00	82,115	18.99	7.9
United States.....	4	8.82	4.38	3.56	49.97	76,258	17.64	7.2
Tobolsk:								
Foreign.....	2	9.68	5.06	3.35	50.68	79,262	18.33	6.8
United States.....	9	10.12	4.08	3.49	50.30	77,252	17.87	6.2
North Finnish Black:								
Foreign.....	3	8.19	4.54	2.75	51.37	78,949	18.26	6.3
United States.....	12	9.44	4.06	3.44	50.56	76,733	17.75	6.7
Shatilov:								
Foreign.....	1	9.41	4.81	3.47	49.93	77,727	17.98	6.8
United States.....	1	9.19	3.43	5.00	46.57	72,410	16.75	6.4
Red Algerian:								
Foreign.....	1	8.24	4.98	2.75	52.70	79,154	18.31	8.1
United States.....	10	10.29	4.32	3.18	49.75	77,036	17.82	6.1
United States.....	164	+	—	+	—	—	—	—
Foreign.....	14	—	+	—	+	+	+	+

a The United States grown Shatilov has less protein than the foreign.

b The United States grown North Finnish Black has a broader nutritive ratio than the foreign.

In considering this table it will be noticed that while in most cases the number of domestic samples was considerably larger than the number of foreign and in two cases only one sample each was analyzed, still the comparison is even more striking than in the preceding table. The general statement that the domestic oats contain more protein and crude fiber and less fat and carbohydrate with a lower total production value and a narrower nutritive ratio is upheld by the results in this table with two exceptions. Of the variety Shatilov, only one sample each of domestic and foreign origin having been analyzed, the domestic grain fell below the foreign grown in protein content though it showed the characteristic composition in all other points. The North Finnish Black domestic-grown oat showed the characteristic distinction from the foreign grown in all the nutritive constituents and in the production value, but in the nutritive ratio it differed by being broader, not narrower, than the foreign-grown grain.

These characteristic differences, as shown by average results, are exemplified in a striking way by some individual cases, though, of course, there are also some marked exceptions. The group averages for protein content of domestic and foreign oats show a higher protein content for the domestic samples (Table XVIII, p. 24), and the complete table of individual data (Table XXXVIII, p. 48) shows that of the 54 foreign-grown samples there is only one that has a protein content above the average of the domestic grain. This is No. 1224, North Finnish Black grown in Finland, with digestible protein per hundred pounds amounting to 12.33 pounds, the average of all United

States samples being 10.73 pounds. Of the 242 domestic samples there are only 58 which fell below the average (9.07 pounds) of foreign-grown oats in protein content, and furthermore there is only one sample out of the 54 foreign-grown oats which has a nutritive ratio as narrow or narrower than the average of the United States samples, namely 5.8. This is the same sample, No. 1224, with a nutritive ratio of 5.1. There are only 39 of the 242 domestic samples which have a nutritive ratio as broad or broader than the average (7.3) of the foreign. Three foreign samples show a production value as low, or lower, than the average United States grain, while there are 18 samples of the United States oats that have a production value as high or higher than the maximum foreign grown.

Of the United States samples the variety which gave the highest average protein content is the Sixty Day oat, 39 samples of which had an average digestible protein content of 11.46 pounds. The highest individual samples were two samples of Sixty Day oat, one grown in Ohio, No. 1297, with 15.40 pounds digestible protein, and the other in South Dakota, No. 1274, with 15.12 pounds. These three instances were also the ones showing the narrowest nutritive ratios, which were, respectively, 5.3, 3.7, and 3.9. The lowest average protein content of the United States grain was found in four samples of Belgian Winter, which gave 8.82 pounds of digestible protein, being 0.25 pound lower than the average foreign oats and only 0.06 pound higher than the four corresponding samples of Belgian Winter grown in Europe. The nutritive ratio of these domestic Belgian Winter samples was the broadest of all of the United States oats, namely, 7.2, only 0.1 lower than the foreign average.

Thus it is evident that the characteristic tendency of the oats grown in the United States, when compared with oats grown in Europe, is toward a higher protein content with a correspondingly lower amount of carbohydrate, resulting in a narrow nutritive ratio combined with a slightly lower production value.

In comparison with the other cereals, oats belong to the energy or muscle-producing grains with high protein and narrow nutritive ratio as contrasted with the fat or heat-producing grains, such as maize, which possess a smaller amount of protein and a broad nutritive ratio. As shown by Tables XVI and XVII, the oats grown in the United States appear to be less inclined toward the maize type and to have their oat characteristics more strongly emphasized than do the same or similar varieties grown in Europe.

TABLE XVIII.—Oats analyses—average results.

[Pounds per hundred pounds of dry matter.]

Number of analyses.	Variety.	Locality.	Protein (N x 6.25).			Fat.			Crude fiber.			Carbohydrate.			Total production value.		Nutritive ratio.
			Total.	Digestible.	Production value. (Gain in flesh.)	Total.	Digestible.	Production value. (Gain in flesh.)	Total.	Digestible.	Production value. (Gain in flesh.)	Total.	Digestible.	Production value. (Gain in flesh.)	Gain in flesh.	Calories.	
54	Miscellaneous.	Foreign.	11.63	7.07	2.13	5.89	4.86	2.96	10.89	2.83	0.70	67.91	32.28	12.96	18.35	79,327	7.3
55	Hull-less.	do.	18.37	14.33	3.37	6.26	3.32	2.74	2.90	7.3	.18	70.05	53.94	13.37	19.66	84,900	4.6
128	Swedish select.	United States.	12.85	10.02	2.35	4.00	3.20	2.15	11.00	3.18	.79	66.99	51.24	12.71	17.68	76,431	6.2
39	Sixty Day.	do.	12.99	11.46	2.69	3.89	3.23	1.70	13.43	2.86	.87	66.55	51.24	12.71	17.81	77,252	5.3
9	Tobolsk.	do.	12.67	10.12	2.38	4.91	4.08	2.18	13.43	3.49	1.24	65.32	46.57	11.55	16.75	72,410	6.4
2	Shatlov.	do.	11.78	9.19	2.16	4.13	3.43	1.80	19.25	3.00	.88	64.90	46.97	12.39	17.64	76,258	7.2
4	Belgian winter.	do.	11.31	8.82	2.07	5.28	4.38	2.30	13.69	3.56	.85	65.67	50.56	12.54	17.75	76,733	6.7
12	North Finnish black.	do.	12.10	9.44	2.22	4.89	4.00	2.14	13.81	4.11	1.02	61.97	47.71	11.83	17.17	74,226	5.8
13	Crown red.	do.	13.25	10.34	2.43	4.33	3.63	1.89	11.60	3.02	.75	65.76	50.62	12.55	18.36	79,370	6.6
8	Rust proof.	do.	12.74	9.94	2.34	4.61	3.15	2.01	11.73	3.05	.76	65.76	50.62	12.55	18.36	79,370	6.6
5	Cullerson winter.	do.	12.74	9.94	2.34	4.61	3.15	2.01	11.73	3.05	.76	65.76	50.62	12.55	18.36	79,370	6.6
10	Red Algerian.	do.	13.19	10.29	2.42	6.20	4.22	2.27	12.25	3.18	.79	64.62	49.75	12.34	17.82	77,036	6.2
7	Burt.	do.	14.34	11.19	2.63	6.21	4.15	2.71	11.41	2.97	.74	63.67	49.02	12.16	18.24	78,552	5.7
2	Snoma.	do.	13.22	10.31	2.42	6.23	4.17	2.72	9.26	2.41	.60	67.85	52.24	12.95	18.69	80,797	6.4
13	Miscellaneous.	do.	12.96	10.11	2.38	4.33	4.01	2.16	12.56	3.27	.81	65.25	50.24	12.46	17.81	76,993	6.2
242	All U. S. varieties.	do.	13.76	10.73	2.52	4.33	3.99	1.89	12.20	3.17	.79	66.29	51.04	12.66	17.86	77,209	5.8
25	Purely domestic.	do.	13.00	10.60	2.49	5.62	4.67	2.45	11.46	2.96	.74	65.18	50.19	12.44	18.11	78,290	6.0
19	Swedish select.	Nebraska.	12.66	9.87	2.32	3.74	3.10	1.93	14.22	3.70	.92	64.94	50.00	12.68	17.57	74,058	6.1
14	do.	South Dakota.	13.13	10.24	2.41	4.48	3.72	1.96	13.13	3.15	.90	66.42	51.14	12.68	17.83	77,079	6.1
14	do.	Iowa.	12.85	8.89	2.37	3.87	3.21	1.69	13.96	3.63	.90	66.50	51.44	12.76	17.44	75,303	7.0
15	do.	Michigan.	12.94	10.09	2.37	4.02	3.41	1.76	11.83	3.08	.90	67.52	52.09	12.92	17.81	77,993	6.2
12	do.	Wisconsin.	11.49	8.96	2.11	4.11	3.59	1.79	12.25	2.49	.92	68.00	52.76	13.08	17.78	76,963	7.1
6	do.	Montana.	13.63	10.63	2.50	4.39	3.69	2.05	9.99	2.49	.62	68.00	52.76	13.08	18.15	78,463	6.0
6	do.	Kansas.	14.46	11.28	2.65	3.95	3.28	1.73	12.12	3.15	.65	68.00	52.76	13.08	18.15	78,463	6.0
5	do.	Washington.	12.66	9.87	2.32	4.45	3.69	1.94	10.09	2.62	.95	69.01	53.14	13.18	18.09	78,202	6.5
5	do.	New York.	13.01	10.15	2.39	3.54	3.23	1.55	11.73	2.88	.71	68.98	53.11	13.13	17.82	77,036	6.2
4	do.	Illinois.	13.44	11.26	2.65	3.74	3.23	1.45	11.79	3.01	.73	67.77	52.18	12.73	17.97	76,041	5.4
4	do.	Pennsylvania.	13.58	10.50	2.49	4.15	3.44	1.81	11.31	2.94	.71	65.87	52.18	12.58	17.66	77,984	5.9
4	do.	Colorado.	14.74	11.50	2.70	3.79	3.15	1.66	11.31	2.90	.84	65.87	52.18	12.58	17.66	77,984	5.9
3	do.	North Carolina.	12.63	9.85	2.31	3.41	2.83	1.49	12.99	3.38	.78	66.94	51.54	12.78	17.42	75,307	6.2

3	do.	7.92	92.06	3.56	13.42	10.47	2.46	3.16	2.62	1.38	11.57	3.01	.75	68.19	52.50	13.02	17.61	76.128	5.9
3	do.	7.76	92.24	4.57	11.53	9.23	2.17	4.61	3.83	2.01	9.92	2.68	.64	68.06	53.17	13.18	18.00	77.814	7.0
2	do.	7.58	92.42	4.15	14.87	11.60	2.73	2.68	3.32	1.45	11.91	3.10	.77	65.73	50.61	12.55	17.50	75.652	5.2
2	do.	8.79	91.21	4.47	11.81	9.21	2.16	2.68	2.21	1.16	15.84	4.12	1.02	62.41	48.05	11.92	16.26	70.292	6.2
2	do.	9.47	90.53	3.57	12.47	9.73	2.29	5.05	4.19	2.20	9.84	2.56	.83	66.56	53.56	13.28	18.40	79.543	6.7
2	do.	7.21	92.79	3.34	13.78	10.75	2.53	4.24	3.52	1.85	10.35	2.69	.67	68.28	52.57	13.04	18.09	78.203	5.9
3	do.	8.50	91.50	3.21	13.79	10.76	2.53	4.04	3.35	1.76	10.66	2.77	.69	68.31	52.60	13.05	18.03	77.944	5.8
126	do.	7.10	92.90	3.90	12.85	10.02	2.35	4.00	3.32	1.75	12.23	3.18	.79	66.99	51.58	12.79	17.68	76.430	6.2
14	Sixty Day	9.05	90.95	3.74	14.96	11.67	2.74	4.25	3.53	1.86	10.71	2.78	.69	66.33	51.07	12.66	17.95	77.598	5.3
4	do.	8.38	91.62	4.08	14.78	11.53	2.71	4.25	3.53	1.86	10.10	2.63	.65	66.77	51.41	12.75	17.97	77.684	5.4
21	do.	8.90	91.10	4.03	14.50	11.31	2.66	3.50	2.98	1.57	11.37	2.96	.73	66.65	51.32	12.73	17.69	76.473	5.4
39	do.	8.90	91.10	3.94	14.69	11.46	2.69	3.89	3.23	1.70	11.00	2.86	.71	66.55	51.24	12.71	17.81	76.993	5.3
7	Tobolsk.	8.44	91.56	3.32	12.44	9.70	2.25	4.96	4.12	2.17	13.89	3.61	.89	65.39	50.35	12.48	17.82	77.036	6.5
2	do.	9.34	90.66	3.54	14.84	11.57	2.72	4.72	3.92	2.06	11.84	3.08	.76	65.05	50.08	12.42	17.96	77.641	5.3
9	do.	8.53	91.47	3.37	12.97	10.12	2.28	4.91	4.08	2.15	13.43	3.49	.87	65.32	50.29	12.47	17.87	77.252	6.2
5	North Finnish black	9.33	90.67	4.03	11.66	9.09	2.14	3.81	3.16	1.66	15.74	4.09	1.01	64.75	49.85	12.36	17.17	74.226	6.7
7	do.	8.35	91.65	3.98	12.42	9.69	2.28	5.07	4.71	2.48	11.59	3.01	.75	66.33	51.08	12.67	18.18	78.592	6.7
12	do.	8.76	91.24	4.00	12.10	9.44	2.22	4.89	4.06	2.14	13.32	3.46	.86	65.67	50.56	12.54	17.76	76.777	6.7
2	Red Algerian	8.98	91.02	4.31	12.78	9.97	2.34	4.95	4.11	2.16	12.99	3.38	.84	64.98	50.05	12.41	17.75	76.712	6.3
8	do.	8.51	91.49	4.85	13.29	10.36	2.44	5.26	4.37	2.30	12.07	3.14	.78	64.53	49.69	12.32	17.84	77.101	6.2
10	do.	8.61	91.39	4.74	13.19	10.29	2.42	5.20	4.32	2.27	12.25	3.18	.79	64.62	49.75	12.34	17.82	77.036	6.2
	All United States.																		
	South Dakota.	9.05	90.95	3.74	14.96	11.67	2.74	4.25	3.53	1.86	10.71	2.78	.69	66.33	51.07	12.66	17.95	77.598	5.3
	North Dakota.	8.38	91.62	4.08	14.78	11.53	2.71	4.25	3.53	1.86	10.10	2.63	.65	66.77	51.41	12.75	17.97	77.684	5.4
	Miscellaneous.	8.90	91.10	4.03	14.50	11.31	2.66	3.50	2.98	1.57	11.37	2.96	.73	66.65	51.32	12.73	17.69	76.473	5.4
	All United States.	8.90	91.10	3.94	14.69	11.46	2.69	3.89	3.23	1.70	11.00	2.86	.71	66.55	51.24	12.71	17.81	76.993	5.3
	South Dakota.	8.44	91.56	3.32	12.44	9.70	2.25	4.96	4.12	2.17	13.89	3.61	.89	65.39	50.35	12.48	17.82	77.036	6.5
	Miscellaneous.	9.34	90.66	3.54	14.84	11.57	2.72	4.72	3.92	2.06	11.84	3.08	.76	65.05	50.08	12.42	17.96	77.641	5.3
	All United States.	8.53	91.47	3.37	12.97	10.12	2.28	4.91	4.08	2.15	13.43	3.49	.87	65.32	50.29	12.47	17.87	77.252	6.2
	South Dakota.	9.33	90.67	4.03	11.66	9.09	2.14	3.81	3.16	1.66	15.74	4.09	1.01	64.75	49.85	12.36	17.17	74.226	6.7
	Miscellaneous.	8.35	91.65	3.98	12.42	9.69	2.28	5.07	4.71	2.48	11.59	3.01	.75	66.33	51.08	12.67	18.18	78.592	6.7
	All United States.	8.76	91.24	4.00	12.10	9.44	2.22	4.89	4.06	2.14	13.32	3.46	.86	65.67	50.56	12.54	17.76	76.777	6.7
	South Dakota.	8.98	91.02	4.31	12.78	9.97	2.34	4.95	4.11	2.16	12.99	3.38	.84	64.98	50.05	12.41	17.75	76.712	6.3
	Miscellaneous.	8.51	91.49	4.85	13.29	10.36	2.44	5.26	4.37	2.30	12.07	3.14	.78	64.53	49.69	12.32	17.84	77.101	6.2
	All United States.	8.61	91.39	4.74	13.19	10.29	2.42	5.20	4.32	2.27	12.25	3.18	.79	64.62	49.75	12.34	17.82	77.036	6.2

PRODUCTIVE VALUE AND NUTRITIVE RATIO AS A BASIS FOR EXACT VALUATION.

From a study of the data presented in these or similar tables of analysis of feeding stuffs, it is seen that by considering simply differences in the amounts of the four nutritive constituents a very indefinite conclusion is often reached. If two distinctly different cereals are considered, such as oats and corn, the differences in the amounts of the nutritive constituents are considerable, as would be expected. When, however, we have to deal with cereals that are very similar, such as rye and wheat, or with simply different samples or varieties of the same cereal, the differences in the amounts of the nutritive constituents are so small and so nearly counterbalance each other that it is impossible to form any opinion as to their relative value based on these factors alone. It is, therefore, necessary to secure some factor that either sums up or magnifies the slight differences in composition if a true valuation of similar feeding stuffs is to be made. Even with such a factor it is natural that differences should not be great between different samples or different varieties of the same cereal. The most distinctly different foreign and domestic oat, for example, can not show wider differences in composition than are possible between plants belonging to the same species.

Various efforts have been made to obtain such a factor, and it has seemed to the writer that the best comparison and relative valuation of similar or slightly different samples of feeding materials is secured by a consideration of the *production value*, and especially when this is considered in connection with the *nutritive ratio*. The production value gives, in the most accurate terms known, the amount of energy (expressed either in calories, weight of flesh gained, or in any other unit), which a definite amount of a certain food will produce when fed as a production ration in addition to a basal maintenance ration. The nutritive ratio gives the relation between the protein and other nutrient constituents of a food, i. e., between the constituents essential in producing muscular energy and those essential in producing fat and heat. As the uses of various foods are different, one class for producing muscular energy or work and the other class for increasing flesh or maintaining animal heat, so the value of a food will depend upon the use for which it is intended, and it is not possible to use any factor for the valuation of foods without considering this point.

Cereal grains; as a class, are naturally much alike in their general composition, but may be divided into two subclasses or groups, the one typified by oats or wheat, high in protein, relatively low in carbohydrate, with a narrow nutritive ratio, and the other, typified by maize, being the opposite of the first, low (relatively) in protein, rich in carbohydrate, and with a broad nutritive ratio.

With these two typical grains as examples we may judge of the tendency of a variety or sample, under certain observed conditions

of growth, etc., to partake of the properties of one or the other of these extremes and in the case of these two cereals themselves to observe whether a variety or sample shows by its composition a more strongly typified individual of its class or whether it is less typical and inclined toward the other class.

TABLE XIX.—*Comparison of oats and maize as typical energy-producing and fat-producing cereals.*

[Pounds per hundred pounds of dry matter.]

Grain.	Digestible nutrients.				Production value.		Nutritive ratio.
	Protein.	Fat.	Crude fiber.	Carbohydrate.	Calories.	Flesh gained.	
Oats.....	10.73	3.59	3.17	51.04	77,209	17.86	5.8
Maize.....	7.14	3.92	1.28	77.86	100,985	23.36	10.5

The production value and the nutritive ratio being the summation or multiplication of the individual differences in composition are, as is so plainly shown in this table, much better adapted to serve as a basis for a relative valuation of the materials than is any one or all of the separate constituents. As has previously been pointed out, however, it can not be said, in this particular case, that because a hundred pounds of maize will produce an increase in flesh 5.5 pounds greater than can be produced with an equal weight of oats that therefore maize is more valuable than oats; or that because the nutritive ratio of oats is 4.7 lower than that of maize it is consequently of less value. It is true that the amount of productive energy in 100 pounds of maize is greater than in an equal amount of oats by some 23,000 Calories and its intrinsic food value is that much greater; but it is not true that, for all purposes, maize is a more valuable food than oats. It is impossible to compare the value of foods distinctly different in their nature and properties solely by means of any of the factors given in this table or previously discussed.

If, however, two samples or varieties of the same cereal or of two cereals very similar, such as rye and wheat, or emmer and einkorn, or proso and maize, are compared, then, from a consideration of these factors, a just and definite idea of their relative value as animal foods may be formed. A combination of the two factors—production value and nutritive ratio—gives the clearest and most trustworthy basis for such valuation. If two samples of oats or two samples of different cereals of the oat type are compared, it may be said that the one possessing the highest production value and the lowest or narrowest nutritive ratio is the most valuable for the feeding purposes for which oats are used, namely, as producers of muscular energy or working strength.

On the other hand, of two samples of maize, the one possessing the highest production value and the highest or broadest nutritive ratio is the most valuable as a fat-producing food. Of course it is true that differences are not always clean cut, so that allowance must often be made, and a difference in one factor may be more than counterbalanced by that in another; but, nevertheless, it is upon a consideration of these two factors that a true and just valuation of animal foods must be based.

In considering, then, the oats that have been under discussion the question now arises: Have these different varieties of foreign grain changed by their introduction into the United States, and, if so, is this change toward a still more strongly typical oat or in the reverse direction? This is without reference to the effect of the change of environment on the physiological properties of resistance to drought and disease, which must be determined by other studies.

In the tables on the comparison of domestic and foreign grown oats (Tables XVI and XVII, pp. 21 and 22) it is shown that the distinct difference between United States and foreign oats is that the former are higher in protein content and possess a narrower nutritive ratio and sometimes a lower production value. The important difference is not, however, in the production value, but in the nutritive ratio. Upon examination of the tables it appears that the differences in the production values are not great, and, though the foreign oats studied have the highest production values that were found, they are not much above the purely domestic oat, the maximum difference being about 0.25 pound of flesh gained per 100 pounds grain fed, or about 1,000 Calories or 1 Therm, according to the new unit of Armsby. The average production value for the five groups of United States oats given in Table XVI, page 21, is 17.87 pounds of flesh gained or 77,266 Calories, while the average of the three groups of foreign oats is only 18.26 pounds of flesh gained, or 78,954 Calories, a difference of only 0.39 pound of flesh gained, or 1,688 Calories. This difference is not large, especially when it is remembered that the purely domestic oats have a mean production value of 18.11 pounds of flesh gained, only 0.15 pound below the foreign average.

The nutritive ratio, however, shows a very noticeable difference. The nutritive ratio of the domestic oats, with one exception, varies from 1:4.6 to 1:6.6, while that of the foreign is 1:7.0 to 1:7.3. This shows clearly, in another way, the fact previously emphasized that the domestic oat is richer in protein. Table XX shows the different varieties of purely United States oats compared with the three groups of foreign oats.

TABLE XX.—*Different varieties of domestic oats compared with foreign oats.*

[Pounds per hundred pounds of dry matter.]

Variety.	No. of samples.	Digestible nutrients.				Production value.		Nutritive ratio.
		Protein.	Fat.	Crude fiber.	Carbo-hydrate.	Flesh gained.	Calories.	
Domestic oats:								
Rust proof.....	8	10.39	3.83	3.02	50.60	17.75	76,733	6.0
Culberson Winter.....	5	9.94	5.15	3.05	50.62	18.36	79,370	6.6
Burt.....	7	11.19	5.15	2.97	49.02	18.24	78,852	5.7
Snoma.....	2	10.31	5.17	2.41	52.24	18.69	80,797	6.4
Prince Edward Island.....	1	13.56	5.17	2.63	47.92	18.44	79,716	4.6
Big Four.....	1	8.77	3.80	3.91	50.37	17.52	75,739	7.2
White Russian.....	1	10.87	5.00	2.78	50.70	18.44	79,716	5.9
Mean.....	25	10.60	4.67	2.98	50.19	18.11	78,377	6.0
Typical U. S. oat, 1898 ^a		10.40	4.15	3.46	49.62	17.88	77,395	6.0
Mean U. S. and Canada, 1893 ^a	72	10.45	4.07	3.44	49.84	17.80	77,049	6.0
Foreign oats:								
Chamberlain.....	54	9.07	4.86	2.83	52.28	18.35	79,327	7.3
Kellner ^a		9.26	4.59	3.09	51.69	18.17	78,510	7.0
König ^a	307	9.07	5.02	2.93	51.57	18.28	79,024	7.2

^a Chamberlain, loc. cit.

These are the results that without question should be used as a basis for the comparison of United States and foreign oats and on which we should decide whether introduced varieties are desirable so far as the effect of the introduction on their composition is concerned. An examination of the three results of (1) a typical United States oat as given in 1898, (2) the mean of 72 United States and Canadian samples in 1893, and (3) the mean of 25 samples of strictly United States oats of 1906 show that the typical oats grown in the United States at the three dates—1893, 1898, and 1906—are very nearly alike, especially in the more important factors—protein, carbohydrate, production value, and nutritive ratio—and that they differ materially from the foreign oats reported in this table.

This shows that the composition of United States oats, as given, is not obtained during one season nor on one variety, but that the average composition of oats grown in such different localities, as shown in the detailed tables given at the close of this bulletin (p. 48) and in different years, is distinctly different in certain points from a similar average composition of foreign oats.

The factor which shows the least variation is the production value. This means that while the amount of the nutritive constituents is different the real nutritive and productive value is only slightly changed. That is, while the food value of the domestic oats remains on the average nearly as high, and in individual instances is higher than the foreign oats, its distinctive character as producing muscle and energy rather than fat is emphasized. Thus, the mean of the purely domestic oats as to production value is only slightly lower than that of the foreign oats, namely, 18.11 pounds of flesh gained, or 78,377 Calories, compared with 18.17 to 18.35 pounds of flesh

gained, or 78,510 to 79,327 Calories. The difference in the nutritive ratios is, however, considerable, namely, 1:6.0 and 1:7.2. The loss, therefore, in production value appears to be more than compensated for by the gain in protein over carbohydrate.

It will be seen that the production value of the typical oat of 1893 and 1898, as well as that of the Rust Proof and Big Four varieties, is considerably lower than that of the other domestic oats and of the foreign oats. The cause of the higher production value of these other varieties is that the amount of fat is high, so that the production value is high, while at the same time the ratio of protein to carbohydrate is low.

If one individual variety, for example, domestic Swedish Select, be closely compared with the foreign seed, the same general relations are found to exist. From Tables XVII and XVIII (pp. 22 and 24) it is seen that in twelve States out of twenty the domestic oats increased in protein content over the original foreign seed, in four cases the production value increased, and in all but five the nutritive ratio became narrower.

From four States the protein was approximately equal to the foreign and only four out of the twenty fell below the foreign. The four States yielding oats containing less protein than the foreign seed were: Iowa (8.89), Wisconsin (8.96), Idaho (9.23), and Missouri (9.21), the foreign seed averaging 9.89. With Tobolsk, North Finnish Black, Belgian Winter, Red Algerian, and Shatilov, the only other varieties of which we have the analysis of the foreign seed from which the domestic grain was grown, the same facts are true, with the exception of Shatilov, as previously mentioned.

CONCLUSIONS.

The general conclusion, therefore, in regard to the oats is that the domestic grain is more strongly typical of its class than the foreign-grown oats, and that on introduction into the United States the foreign varieties change somewhat in composition, but this change is toward rather than away from the typical oat—this typical oat being characterized by high protein and narrow nutritive ratio, a grain essentially productive of muscular strength.

EMMER (*Triticum dicoccum*) AND EINKORN (*Triticum monococcum*).

These two cereals, emmer (*Triticum dicoccum*) and einkorn (*Triticum monococcum*), belong botanically to the same genus as wheat, but on account of the fact that they are both used as food without the removal of the hull they may better be compared with oats. The large amount of crude fiber reduces the protein and carbohydrate below that of wheat and lowers the production value even to less than that of oats. The nutritive ratio for einkorn likewise falls below that of oats, being as low as 1:5.2. Einkorn is more fibrous

than emmer, and has more crude fiber, a lower production value, and a narrower nutritive ratio. Table XXI shows a comparison of emmer, einkorn, and oats.

TABLE XXI.—*Comparison of the total and digestible nutrients of emmer, einkorn, and oats.*

[Pounds per hundred pounds of dry matter.]

Cereal.	Nutrients.								Total production value.		Nutri- tive ratio.
	Protein.		Fat.		Crude fiber.		Carbohydrate.		Gain in flesh.	Calo- ries.	
	Total.	Digest- ible.	Total.	Digest- ible.	Total.	Digest- ible.	Total.	Digest- ible.			
Emmer.....	13.28	9.96	1.91	1.36	11.31	4.98	69.42	52.06	17.20	74,356	6.0
Einkorn.....	14.67	11.00	2.19	1.55	13.55	5.96	64.02	48.04	16.81	72,670	5.2
Oats.....	13.76	10.73	4.33	3.59	12.20	3.17	66.29	51.04	17.86	77,209	5.8

The comparison of these three cereals will be discussed more fully in the general conclusion. There being only one sample of foreign emmer no comparison of domestic and foreign grain of these two varieties is possible. The highest protein content of the emmers was in a sample from South Dakota, 1903, No. 1051, having 18.69 pounds of total protein and 14.02 pounds of digestible protein per hundred pounds, the average protein being 13.28 pounds total and 9.96 pounds digestible.

In the case of the einkorn, No. 1065, a sample from Kansas, 1905, had the highest protein content, a total of 16.25 pounds and 12.19 pounds of digestible protein per hundred pounds.

TABLE XXII.—*Emmer and einkorn analyses—average results.*

[Pounds per hundred pounds of dry matter.]

Number of analyses.	Variety.	Locality.				Protein (N×6.25).		
			Water.	Dry matter.	Ash.	Total.	Digestible.	Production value—gain in flesh.
1	Emmer:							
24	Black emmer.....	Foreign.....	8.92	91.08	3.88	12.00	9.00	2.11
	Do.....	United States.....	8.68	91.32	4.07	13.28	9.96	2.34
4	Einkorn: Miscellaneous.....	do.....	8.34	91.66	5.57	14.67	11.00	2.58
Number of analyses.	Variety.	Locality.	Fat.			Crude fiber.		
			Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.
1	Emmer:							
24	Black emmer.....	Foreign.....	1.47	1.04	0.55	10.72	4.72	1.17
	Do.....	United States.....	1.91	1.36	.71	11.31	4.98	1.24
4	Einkorn: Miscellaneous.....	do.....	2.19	1.55	.81	13.55	5.96	1.48

TABLE XXII.—*Emmer and einkorn analyses—average results—Continued.*

Number of analyses.	Variety.	Locality.	Carbohydrate.			Total production value.		Nutritive ratio.
			Total.	Digestible.	Production value—gain in flesh.	Gain in flesh.	Calories.	
1	Emmer:							
24	Black emmer.....	Foreign.....	71.93	53.95	13.38	17.21	74,399	6.8
4	Do.....	United States.....	69.42	52.06	12.91	17.20	74,356	6.0
	Einkorn: Miscellaneous.....	do.....	64.02	48.04	11.94	16.81	72,670	5.2

WHEAT (*Triticum vulgare*).

The wheat samples analyzed for this investigation comprise only a small number representing the principal classes of American wheat and a few samples of American and foreign durum. In explanation of the seeming neglect of this important cereal, it may be said that the primary object of this study was the analysis of oats, barley, rye, emmer, einkorn, proso, and sorghum; wheat and maize were only introduced when it became desirable to report the chemical results alone in the form of a bulletin. Furthermore, there have been many extended series of analyses made of both of these cereals, and for the purposes of comparison such results can readily be used.

The digestible nutrients in the wheat samples analyzed and in typical groups as taken from other sources are given in Table XXIII.

TABLE XXIII.—*Wheat—digestible nutrients, production value, and nutritive ratio.*

[Pounds per hundred pounds of dry matter.]

Description of samples.	Number of samples.	Digestible nutrients.				Production value.		Nutritive ratio.
		Protein.	Fat.	Crude fiber.	Carbohydrates.	Calories.	Flesh gained.	
Foreign durum.....	3	14.17	1.42	1.13	70.56	94,501	21.96	5.3
Domestic durum.....	12	13.02	1.72	1.33	70.77	94,457	21.85	5.8
Northwest Spring, United States.....	4	11.78	1.41	1.17	73.04	94,717	21.91	6.6
Kansas Winter, United States.....	3	11.06	1.14	1.31	73.55	94,068	21.76	7.0
Soft Winter, United States.....	5	9.96	1.20	1.17	75.24	94,760	21.92	7.9
United States wheat, 1893 ^a	166	10.48	1.25	1.24	74.27	94,460	21.85	7.5
Canada wheat, 1893 ^a	49	10.62	1.28	1.20	74.28	94,635	21.89	7.4
English wheat (König) ^b	15	9.72	2.15	1.57	74.43	96,235	22.26	8.3
Russian wheat (König) ^c	33	14.82	1.15	1.19	69.85	93,809	21.70	4.9
Germany Spring (König) ^d	30	13.22	1.13	* 1.53	* 62.75	84,905	19.64	5.0
Winter (König) ^d	42	10.87	1.24	1.53	63.99	84,080	19.45	6.3
Typical United States wheat ^e		10.50	1.23	1.26	73.33	93,465	21.62	7.4

^a U. S. Dept. Agr., Bureau of Chemistry, 1895, Bul. 45, pp. 45, 47.^b Loc. cit., p. 422.^c Loc. cit., p. 421.^d Loc. cit., p. 418, 419.^e Calculated.^f U. S. Dept. Agr., Bureau of Chemistry, 1898, Bul. 13, Pt. IX, p. 1189.

No direct comparison can be made between foreign and domestic wheat, as was done with both oats and barley, because the domestic grain was not the product of directly introduced foreign wheat. In the case of the durum wheat this was true in a general way, but not as with the other cereals.

It will be readily seen that the highest protein wheats are those from Russia, while the spring wheats of North Germany and the durum and spring wheats of the United States rank next. While the highest production value occurs in the English wheat which is very low in protein, all of the hard wheats high in protein are likewise high in total production value. The actual production value is much higher for wheat than for any of the other energy-producing cereals. This is readily explained because of the small amount of crude fiber present, almost the entire amount of nitrogen-free extract constituents being in the form of true carbohydrate which possesses a relatively high coefficient of digestibility.

While the effect of introduction on European oats and barley is to increase the protein content, the effect produced on Russian durum wheat, which is originally a very hard, high protein wheat, is, at least in most localities, to lower the protein content to some extent. Other results, however, show that in the drier semiarid regions of the United States the protein content even of Russian durum wheat is maintained or may even be increased, so that this is not really any exception to the general effect of introduction on foreign-grown cereals.

The hard wheats, possessing, as they do, a large amount of protein, have a narrow nutritive ratio, so that taken together with the high production value they are naturally the most concentrated of the energy-producing cereals of the oat class.

TABLE XXIV.—*Wheat analyses—average results.*

[Pounds per hundred pounds of dry matter.]

Number of analyses.	Variety.	Locality.	Water.	Dry matter.	Ash.	Protein (N×5.7).		
						Total.	Digestible.	Production value—gain in flesh.
3	Durum.....	Russia.....	11.02	88.98	1.78	16.87	14.17	3.33
1	<i>T. vulgare</i>	do.....	9.43	90.57	1.85	16.76	14.07	3.31
1	<i>T. sativum</i>	India.....	9.58	90.42	2.30	11.97	10.05	2.36
12	Durum.....	United States.....	9.77	90.23	2.02	15.50	13.02	3.06
4	Northwest spring.....	do.....	10.11	89.89	1.85	14.02	11.78	2.77
3	Kansas winter.....	do.....	10.68	89.32	2.28	13.17	11.06	2.60
5	Soft winter.....	do.....	10.55	89.45	2.00	11.86	9.96	2.34
24	All varieties.....	do.....	10.10	89.90	2.02	14.20	11.93	2.80

Number of analyses.	Variety.	Locality.	Fat.			Crude fiber.		
			Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.
3	Durum.....	Russia.....	2.25	1.42	0.75	2.40	1.13	0.28
1	<i>T. vulgare</i>	do.....	1.72	1.08	.57	2.45	1.15	.28
1	<i>T. sativum</i>	India.....	2.62	1.65	.87	3.06	1.44	.36
12	Durum.....	United States.....	2.73	1.72	.91	2.83	1.33	.33
4	Northwest spring.....	do.....	2.24	1.41	.74	2.50	1.17	.29
3	Kansas winter.....	do.....	1.81	1.14	.60	2.79	1.31	.32
5	Soft winter.....	do.....	1.91	1.20	.63	2.49	1.17	.29
24	All varieties.....	do.....	2.36	1.49	.78	2.78	1.30	.32

TABLE XXIV.—*Wheat analyses—average results—Continued.*

Number of analyses.	Variety.	Locality.	Carbohydrate.			Total production value.		Nutritive ratio.
			Total.	Digestible.	Production value—gain in flesh.	Gain in flesh.	Calories.	
3	Durum.....	Russia	76.70	70.56	17.50	21.86	94,501	5.3
1	<i>T. vulgare</i>	do.	77.22	71.04	17.62	21.76	94,068	5.3
12	<i>T. sativum</i>	India	80.05	73.65	18.26	21.85	94,467	7.8
1	Durum.....	United States.	76.92	70.77	17.55	21.85	94,457	5.8
4	Northwest spring.....	do.	79.39	73.04	18.11	21.91	94,717	6.6
3	Kansas winter.....	do.	79.95	73.55	18.24	21.76	94,068	7.0
5	Soft winter.....	do.	81.77	75.24	18.66	21.92	94,760	7.9
24	All varieties.....	do.	78.72	72.42	17.96	21.86	94,515	6.5

RYE (*Secale cereale*).

As a feeding grain rye is very similar to wheat, the two being more nearly alike in the essential points, namely, protein content, production value, and nutritive ratio, than any other two cereals.

There are no foreign varieties among the samples of rye analyzed in this study, so that a direct comparison of the introduced varieties with the same varieties grown abroad was impossible. From other analyses made in this country and from analyses reported by König a comparison somewhat similar to that made with oats and barley is possible. In Table XXV the results obtained in this study and those obtained in 1893 and 1898 by the Bureau of Chemistry are compared with foreign rye, the analyses of which are reported by König:

TABLE XXV.—*Rye—total and digestible nutrients, production value, and nutritive ratio.*

[Pounds per hundred pounds of dry matter.]

Description of samples.	Number of samples.	Nutrients.								Production value.		Nutritive ratio.
		Protein.		Fat.		Crude fiber.		Carbohydrate.		Gain in flesh.	Calories.	
		Total.	Digestible.	Total.	Digestible.	Total.	Digestible.	Total.	Digestible.			
Domestic:												
All varieties, 1906.....	17	13.44	11.29	1.83	1.17	2.30	1.22	80.24	73.82	21.87	94,544	6.9
Typical United States, 1898.....		13.09	11.50	1.67	1.07	2.35	1.25	80.17	73.76	21.86	94,502	6.7
All United States, 1893.....	18	13.91	11.68	1.85	1.18	2.34	1.24	79.85	73.46	21.90	94,680	6.6
Foreign: German (König).....	119	12.89	10.83	1.88	1.20	3.02	1.60	79.78	73.40	21.78	94,178	7.2

^a U. S. Dept. Agr., Bureau of Chemistry, 1898, Bul. 13, Pt. IX, p. 1184.^b U. S. Dept. Agr., Bureau of Chemistry, 1893, Bul. 45, p. 37.^c Loc. cit., p. 474.

While the foreign and domestic ryes in Table XXV have no direct relation to each other, as in the case of the oats and barley, and while the differences are not large, it will be noticed that the same general relation exists here as with the other cereals, namely, that the United

States grain is higher in protein than the foreign. In the case of rye, the production value also is higher. The domestic samples, also, though analyzed in three different years, agree more closely with each other than they do with the foreign-grown grain.

From an examination of Table XLII, page 57, it will be seen that the rye with the highest protein content is a sample of Ivanov, grown in Kansas in 1905 (No. 1070), the total protein amounting to 17.44 pounds per 100 pounds, or 14.65 pounds digestible. The three samples with the highest protein content represent each of the varieties examined and were grown in the States of Kansas and Nebraska, the total protein in these three samples being for No. 1070, 17.44 pounds; for No. 1082, 16.44 pounds; and for No. 1076, 15.62 pounds per hundred pounds. The digestible protein equals 14.65 pounds, 13.81 pounds, and 13.12 pounds, respectively. The nutritive ratios of these samples are the narrowest of all the samples, being 1 : 5.0, 5.4, and 5.8, respectively.

TABLE XXVI.—*Rye analyses—average results.*

[Pounds per hundred pounds of dry matter.]

Number of analyses.	Variety	Locality.	Water.	Dry matter.	Ash.	Protein (N×6.25).		
						Total.	Digestible.	Production value—gain in flesh.
6	Ivanov.....	United States.	9.39	90.61	2.15	12.88	10.82	2.54
5	Abbruzzes.....	do.	9.18	90.82	2.23	13.46	11.31	2.66
5	Spring.....	do.	9.58	90.42	2.14	14.18	11.91	2.80
1		do.	9.37	90.63	2.27	13.06	10.97	2.58
17	All varieties.....	do.	9.38	90.62	2.18	13.44	11.29	2.65

Number of analyses.	Variety	Locality.	Fat.			Crude fiber.		
			Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.
6	Ivanov.....	United States.	1.84	1.18	0.62	2.39	1.27	0.31
5	Abbruzzes.....	do.	1.83	1.17	.61	2.24	1.19	.29
5	Spring.....	do.	1.82	1.16	.61	2.23	1.18	.29
1		do.	1.84	1.18	.62	2.47	1.31	.32
17	All varieties.....	do.	1.83	1.17	.61	2.30	1.22	.30

Number of analyses.	Variety.	Locality.	Carbohydrate.			Total production value.		Nutritive ratio.
			Total.	Digestible.	Production value—gain in flesh.	Gain in flesh.	Calories.	
6	Ivanov.....	United States.	80.73	74.27	18.42	21.89	94,630	7.2
5	Abbruzzes.....	do.	80.24	73.82	18.31	21.87	94,544	6.9
5	Spring.....	do.	79.63	73.26	18.17	21.87	94,544	7.1
1		do.	80.36	73.93	18.33	21.85	94,457	7.1
17	All varieties.....	do.	80.24	73.82	18.31	21.87	94,544	6.9

BARLEY (*Hordeum vulgare*).

The barleys which were studied in this investigation comprise 8 samples of imported grain representing four varieties and 123 samples of grain grown in the United States representing 16 varieties. A comparison of the composition of these various samples with that of other compiled analyses is shown in Table XXVII.

TABLE XXVII.—Comparison of barley analyses by groups, domestic and foreign.

[Pounds per hundred pounds of dry matter.]

Description of samples.	Number of samples.	Moisture.	Ash.	Protein.	Fat.	Crude fiber.	Carbo-hydrate.
Typical United States, 1898 ^a		10.85	2.80	12.34	2.52	4.32	78.02
United States and Canadian, 1893 ^b	32	10.80	2.74	11.98	2.39	4.54	78.35
North and middle Germany ^c	98	12.95	3.52	11.50	2.15	4.96	77.97
Foreign samples, 1906	8	9.38	2.81	12.52	2.06	4.58	78.03
United States, 1906	123	9.32	3.04	13.39	1.87	5.64	76.05
Hull-less, 1906	5	9.07	2.36	16.09	1.90	2.64	77.01

^a U. S. Dept. Agr., Bureau of Chemistry, 1898, Bul. 13, Pt. IX, p. 1173.^b U. S. Dept. Agr., Bureau of Chemistry, 1905, Bul. 45, p. 14.^c König, *Chemie der menschlichen Nahrungs- und Genussmittel*, 1903, p. 508 (recalculated to dry basis).

A difference at once observed on examining this table is that the barleys grown in the United States in 1906 have a higher protein content than the foreign barleys analyzed at the same time. This is also true, to a less degree, of the United States barleys of 1893 and 1898 compared with the analyses reported by König.

It would naturally be expected that the United States barleys would be higher in protein because in this country high protein barleys are considered the best for brewing purposes, while in Europe the best brewing barleys are considered to be those low in protein. Although the barleys studied in this investigation belong to the feeding class rather than to the brewing, the tendency in this country to grow for high protein and in Europe for low protein would have an effect on both feeding and brewing barleys in general. The digestible nutrients, production values, and nutritive ratios of these barleys is shown in Table XXVIII.

TABLE XXVIII.—Barley—digestible nutrients averaged by groups, domestic and foreign.

[Pounds per hundred pounds of dry matter.]

Description of samples.	Number of samples.	Digestible nutrients.				Production value.		Nutritive ratio.
		Protein.	Fat.	Crude fiber.	Carbo-hydrate.	Calories.	Flesh gained.	
Typical United States		8.64	2.24	1.42	71.78	92,339	21.36	9.0
United States and Canada, 1893	32	8.39	2.13	1.50	72.08	92,253	21.34	9.3
Foreign (König)	98	8.05	1.91	1.60	71.73	91,086	21.07	9.6
Foreign, 1906	8	8.76	1.83	1.51	71.78	91,604	21.19	8.8
United States, 1906	123	9.37	1.66	1.86	69.96	90,264	20.88	8.0
Hull-less	5	11.26	1.69	.87	70.85	92,166	21.32	6.7

The same general facts are again shown as were found in the case of the oats, namely, the foreign-grown grain is poorer in protein and richer in carbohydrate; higher in production value and broader in nutritive ratio. A comparison of individual varieties (Table XXIX), of which both foreign and domestic samples were analyzed, shows the same relations to exist.

TABLE XXIX.—*Comparison of domestic and foreign barley, averaged by varieties.*

[Pounds per hundred pounds of dry matter.]

Variety.	Number of samples.	Nutrients.								Production value.		Nutritive ratio.
		Protein.		Fat.		Crude fiber.		Carbohy- drate.		Calories.	Fat gained.	
		Total.	Digesti- ble.	Total.	Digesti- ble.	Total.	Digesti- ble.	Total.	Digesti- ble.			
Black Arabian:												
Foreign.....	5	12.52	8.76	2.11	1.88	4.79	1.58	77.82	71.59	91,604	21.19	8.8
United States...	6	14.04	9.83	2.03	1.81	4.92	1.62	75.84	69.77	90,610	20.96	7.7
Swan Neck:												
Foreign.....	1	13.94	9.76	2.18	1.94	4.06	1.34	76.78	70.63	91,475	21.16	7.8
United States...	5	14.49	10.14	2.20	1.96	4.52	1.49	76.06	69.97	91,345	21.13	7.5
Kitzing:												
Foreign.....	1	11.82	8.27	1.94	1.73	4.15	1.37	79.24	72.90	91,960	21.27	9.4
United States...	2	15.25	10.67	2.00	1.78	4.36	1.44	75.56	69.51	90,912	21.03	7.0
Fan:												
Foreign.....	1	11.75	8.22	1.77	1.58	4.50	1.48	79.14	72.81	91,604	21.19	9.5
United States...	1	14.62	10.23	1.79	1.59	5.04	1.66	75.52	69.48	90,264	20.88	8.3

In every case where the same variety was grown in its original foreign home and then later in the United States it increased in protein and the nutritive ratio became narrower, while the production value decreased very slightly. The resulting effect, therefore, is the production of a better feeding barley than the original.

An examination of the table of averages for barley (Table XXXI) will show that the maximum protein content was found in the two United States samples of Kitzing, namely, total, 15.25 pounds per 100 pounds, or 10.67 pounds digestible, the carbohydrate in the same sample being 75.56 pounds total and 69.51 pounds digestible. The lowest protein content, on the other hand, was found in the eight foreign-grown samples, namely, 12.52 pounds total and 8.76 pounds digestible, the carbohydrate in the same samples being 78.03 pounds total and 71.78 pounds digestible. The production value of the United States Kitzing was 21.03 pounds of flesh gained or 90,912 Calories and of the foreign samples 21.19 pounds of flesh gained or 91,604 Calories, the nutritive ratio being 1:7.0 for the domestic and 1:8.8 for the foreign. It is noticeable that in this variety (Kitzing) the protein content is the highest of all, while at the same time the production value is only slightly lower than that of the foreign-grown grain. The same can be said in general of the varieties Swan Neck, Hanna, and Princess. This means that these

varieties are especially adapted for use as feeding barleys, having a high protein content, a narrow nutritive ratio, and at the same time a high production value.

Table XXX gives the varieties of barley considered in the order of their protein content, together with their production values and nutritive ratios.

TABLE XXX.—*Barleys arranged in order of protein content.*

[Pounds per hundred pounds of dry matter.]

Variety.	Protein.	Production value, flesh gained.	Nutritive ratio.
Hull-less.....	16.09	21.32	6.7
Kitzing.....	15.25	(5) ^a 21.03	7.0
Swan Neck.....	14.49	(2) 21.13	7.5
Black Arabian.....	14.04	(7) 20.96	7.7
Black Smyrna.....	13.98	(8) 20.85	7.6
Hanna.....	13.82	(3) 21.04	7.8
Manchuria.....	13.44	(11) 20.68	7.9
Princess.....	13.42	(4) 21.04	8.1
Tell.....	13.08	(12) 20.65	8.1
Beldi.....	12.92	(10) 20.69	8.3
Tennessee Winter.....	12.84	(9) 20.81	8.4
White Smyrna.....	12.49	(6) 21.02	8.8
Foreign: Black Arabian.....	12.52	(1) 21.19	8.8

^a Figures in parentheses express order according to production value.

It will be noticed that the order of the nutritive ratio is exactly the reverse of that of the protein content; that is, the higher the protein content the lower or narrower the nutritive ratio. This is, of course, as would be expected. In regard to the production value, while it is true that the foreign samples with low protein and correspondingly high carbohydrate have the higher production value, some of the domestic samples with high protein have also a high production value. This is due to the fact that the fat content is high or the crude fiber low and is especially noticeable with the varieties Kitzing, Swan Neck, Hanna, and Princess.

Some individual cases may be noticed showing the maximum and minimum limits in protein content, etc. The highest protein content of all individual samples, excepting the hull-less varieties, is found in No. 1102, Hanna, South Dakota, 1904, third crop after introduction, total protein, 19.94 pounds per 100 pounds; digestible protein, 13.61 pounds. The production value of this sample is 20.59 pounds flesh gained and the nutritive ratio is 1:5.2. The lowest protein content occurs in the case of No. 1185, Princess, California, 1902, first crop after introduction, the total protein amounting to 8.44 pounds, and the digestible protein to 5.91 pounds, the production value being 21.32 pounds of flesh gained and the nutritive ratio 1:13.6. The highest production value is found for No. 1215, Giant Winter, Oregon, 1904, second crop after introduction, the production value being

21.72. The nutritive ratio in this case is 1:12.4 and the digestible protein amounts to 6.56 pounds. No. 1165, Beldi, South Dakota, 1905, third crop after introduction, shows the lowest production value, namely 20.24 pounds of flesh gained, the nutritive ratio in this case being 1:6.2, and the digestible protein, 11.37 pounds. From these cases and from an examination of the complete table (Table XLIII, p. 58) it will be seen that, as has been stated, a high protein usually corresponds to a low production value and a narrow nutritive ratio. In the cases just cited the barley having the highest protein content has a production value only a little higher than the lowest and, vice versa, the one having the highest production value has a protein content only slightly higher than the lowest.

Therefore, for the best feeding barleys, it is essential to secure in one sample or variety a high protein and narrow nutritive ratio, while at the same time the production value remains high. For barleys, then, as for oats, the general conclusion is that the effect of introduction appears to be to increase the protein content and to narrow the nutritive ratio. At the same time the total production value is slightly diminished, but in even a less degree than in the case of oats.

The effect of the introduction has been therefore to improve the barleys as a feeding grain, the tendency being toward the production of those characters, mentioned in the preceding paragraph, which are essential to the best barleys for such purposes.

TABLE XXXI.—*Barley analyses—average results.*

[Pounds per hundred pounds of dry matter.]

Number of analyses.	Variety.	Locality.	Water.	Dry matter.	Ash.	Protein ($N \times 6.25$).		
						Total.	Digestible.	Production value—gain in flesh.
5	Black Arabian.....	Foreign.....	9.43	90.57	2.75	12.52	8.76	2.06
3	Miscellaneous.....	do.....	9.30	90.70	2.91	12.50	8.75	2.06
8	Average.....		9.38	90.62	2.81	12.52	8.76	2.06
6	Black Arabian.....	United States.	9.59	90.41	3.16	14.04	9.83	2.31
32	Hanna.....	do.....	9.44	90.56	2.97	13.82	9.67	2.27
23	Tennessee winter.....	do.....	9.50	90.50	3.17	12.84	8.99	2.11
5	Swan Neck.....	do.....	9.30	90.70	2.74	14.49	10.14	2.38
2	Kitzling.....	do.....	9.35	90.65	2.83	15.25	10.67	2.51
12	Beldi.....	do.....	9.05	90.95	3.15	12.92	9.04	2.12
10	Black Smyrna.....	do.....	9.55	90.45	3.05	13.98	9.79	2.30
4	Princess.....	do.....	9.55	90.45	2.85	13.42	9.39	2.21
12	Telli.....	do.....	8.89	91.11	3.01	13.08	9.16	2.15
10	White Smyrna.....	do.....	9.06	90.94	2.95	12.49	8.74	2.05
7	Miscellaneous.....	do.....	8.98	91.02	3.15	13.18	9.23	2.17
123	Average.....		9.32	90.68	3.04	13.39	9.37	2.20
5	Hull-less.....	United States.	9.07	90.93	2.36	16.09	11.26	2.65

TABLE XXXI.—*Barley analyses—average results—Continued.*

Number of analyses.	Variety.	Locality.	Fat.			Crude fiber.		
			Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.
5	Black Arabian.....	Foreign.....	2.11	1.88	0.99	4.79	1.58	0.39
3	Miscellaneous.....	do.....	1.96	1.74	.92	4.24	1.40	.35
8	Average.....		2.06	1.83	.96	4.58	1.51	.37
6	Black Arabian.....	United States.....	2.03	1.81	.95	4.92	1.62	.40
32	Hanna.....	do.....	1.97	1.75	.92	4.66	1.54	.38
23	Tennessee winter.....	do.....	1.78	1.58	.83	6.02	1.99	.49
5	Swan Neck.....	do.....	2.20	1.96	1.03	4.52	1.49	.37
2	Kitzing.....	do.....	2.00	1.78	.94	4.36	1.44	.36
12	Beldi.....	do.....	1.90	1.69	.89	7.11	2.35	.59
10	Black Smyrna.....	do.....	1.88	1.67	.88	5.68	1.87	.47
4	Princess.....	do.....	1.73	1.54	.81	4.67	1.54	.38
12	Telli.....	do.....	1.68	1.50	.79	7.12	2.35	.58
10	White Smyrna.....	do.....	1.85	1.65	.87	5.32	1.76	.44
7	Miscellaneous.....	do.....	1.66	1.48	.78	6.58	2.17	.54
123	Average.....		1.87	1.66	.87	5.64	1.86	.46
5	Hull-less.....	United States.....	1.90	1.69	.89	2.64	.87	.21

Number of analyses.	Variety.	Locality.	Carbohydrate.			Total production value.		Nutritive ratio.
			Total.	Digestible.	Production value—gain in flesh.	Gain in flesh.	Calories.	
5	Black Arabian.....	Foreign.....	77.82	71.59	17.75	21.19	91,604	8.8
3	Miscellaneous.....	do.....	78.39	72.12	17.88	21.21	91,690	8.8
8	Average.....		78.03	71.78	17.80	21.19	91,604	8.8
6	Black Arabian.....	United States.....	75.84	69.77	17.30	20.96	90,610	7.7
32	Hanna.....	do.....	76.57	70.44	17.47	21.04	90,956	7.8
23	Tennessee winter.....	do.....	76.19	70.10	17.38	20.81	89,962	8.4
5	Swan Neck.....	do.....	76.06	69.97	17.35	21.13	91,345	7.5
2	Kitzing.....	do.....	75.56	69.51	17.24	21.03	90,912	7.0
12	Beldi.....	do.....	74.91	68.92	17.09	20.69	89,443	8.3
10	Black Smyrna.....	do.....	75.40	69.36	17.20	20.85	90,134	7.6
4	Princess.....	do.....	77.32	71.13	17.64	21.04	90,956	8.1
12	Telli.....	do.....	75.10	69.09	17.13	20.65	89,270	8.1
10	White Smyrna.....	do.....	77.39	71.20	17.66	21.02	90,869	8.8
7	Miscellaneous.....	do.....	75.34	69.31	17.19	20.68	89,400	8.1
123	Average.....		76.05	69.96	17.35	20.88	90,264	8.0
5	Hull-less.....	United States.....	77.01	70.85	17.57	21.32	92,106	6.7

PROSO (*Panicum miliaceum*) AND SORGHUM (*Andropogon sorghum*).

These two grains, the proso or broom corn millet and the non-saccharine sorghums, may be considered together, as were the emmer and einkorn. They, however, differ more from each other than do the latter grains because of the considerable amount of crude fiber in the proso and the small amount in the sorghum. This gives to the sorghum a higher carbohydrate content and a higher production value. In protein content and in nutritive ratio, however, they are very similar, and in these and also in production value they both have the general characteristics of maize and belong to the same class.

TABLE XXXII.—Group averages of total and digestible nutrients of proso, sorghum, and maize.

[Pounds per hundred pounds of dry matter.]

Cereal.	Nutrients.								Total production value.		Nutritive ratio.
	Protein.		Fat.		Crude fiber.		Carbohydrate.		Gain in flesh.	Calories.	
	Total.	Digestible.	Total.	Digestible.	Total.	Digestible.	Total.	Digestible.			
Proso.....	12.77	6.28	3.27	2.51	8.95	6.11	71.23	60.69	19.37	83,736	11.5
Sorghum.....	11.71	7.61	3.25	2.27	1.80	.92	81.58	74.24	21.62	93,463	10.5
Maize.....	9.91	7.14	4.40	3.92	2.21	1.28	81.96	77.86	23.36	100,985	12.3

Proso and sorghum, like maize, are lower in protein and higher in carbohydrate, giving higher production value and broader nutritive ratio than the other cereals and thus stand at the opposite extreme from oats.

Of the prosos (Table XLIV), the highest protein content is found for No. 1022 from Oklahoma, namely, 16.87 pounds, digestible protein 8.30 pounds per 100 pounds, giving to this sample the narrowest nutritive ratio (1:8.2) and the lowest production value, 18.70 pounds gain in flesh. The highest carbohydrate content was found in a Colorado sample, No. 1029, with 75.22 pounds total and 64.09 pounds digestible. This gives a production value of 19.76 pounds gain in flesh, which is the highest with one exception, No. 1016, for which the nutritive ratio is 1:14.6, the broadest of the entire list. The average protein content is 12.77 pounds total and 6.28 pounds digestible. The average production value and nutritive ratio are, respectively, 19.37 pounds gain in flesh, or 83,736 Calories, and 1:11.5.

Of the sorghums the highest protein content is in No. 771, which has 14.50 pounds total and 9.42 pounds digestible, the nutritive ratio being relatively narrow, namely, 1:8.2. The highest carbohydrate content is found in No. 770 with 85.31 pounds total and 77.64 pounds digestible. This sample has a production value of 21.71 pounds gain in flesh, the maximum being 21.87 pounds in No. 781. It has the broadest nutritive ratio of any of the samples examined, namely, 1:14.3. The average composition of the sorghum samples is given in Table XXXIII.

TABLE XXXIII.—*Proso, sorghum, and maize analyses—average results for the United States.*

[Pounds per hundred pounds of dry matter.]

Number of analyses.	Variety.	Locality.	Water.	Dry matter.	Ash.	Protein (N × 6.25).		
						Total.	Digestible.	Production value—gain in flesh.
	Proso:							
17	Red Orenburg.....	United States.	8.98	91.02	3.59	12.84	6.32	1.49
9	Red Voronezh.....	do.	8.83	91.17	4.03	13.08	6.43	1.51
10	Black Voronezh.....	do.	8.80	91.20	3.82	12.27	6.04	1.42
2	Yellow Manitoba.....	do.	9.54	90.46	4.10	13.37	6.58	1.55
38	Average.....	do.	8.93	91.07	3.78	12.77	6.28	1.48
10	Sorghum: Miscellaneous.....	do.	11.71	88.29	1.75	11.71	7.61	1.79
12	Maize: Miscellaneous.....	do.	13.06	86.94	1.52	9.91	7.14	1.68

Number of analyses.	Variety.	Locality.	Fat.			Crude fiber.		
			Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.
	Proso:							
17	Red Orenburg.....	United States.	3.29	2.53	1.33	8.56	5.85	1.45
9	Red Voronezh.....	do.	3.09	2.38	1.25	9.72	6.64	1.65
10	Black Voronezh.....	do.	3.33	2.56	1.35	8.84	6.04	1.50
2	Yellow Manitoba.....	do.	3.52	2.71	1.43	9.32	6.37	1.58
38	Average.....	do.	3.27	2.51	1.32	8.95	6.11	1.52
10	Sorghum: Miscellaneous.....	do.	3.25	2.27	1.19	1.80	.92	.23
12	Maize: Miscellaneous.....	do.	4.40	3.92	2.06	2.21	1.28	.32

Number of analyses.	Variety.	Locality.	Carbohydrate.			Total production value.		Nutritive ratio.
			Total.	Digestible.	Production value—gain in flesh.	Gain in flesh.	Calories.	
	Proso:							
17	Red Orenburg.....	United States.	71.71	61.10	15.15	19.42	83,953	11.5
9	Red Voronezh.....	do.	70.09	59.71	14.81	19.22	83,088	11.2
10	Black Voronezh.....	do.	71.74	61.12	15.16	19.43	83,996	12.1
2	Yellow Manitoba.....	do.	69.68	59.38	14.72	19.28	83,347	10.9
38	Average.....	do.	71.23	60.69	15.05	19.37	83,736	11.5
10	Sorghum: Miscellaneous.....	do.	81.58	74.24	18.41	21.62	93,463	10.5
12	Maize: Miscellaneous.....	do.	81.96	77.86	19.31	23.36	100,985	12.3

MAIZE (*Zea mays*).

The analyses of maize given in the tables were made for another study, but they have been used here for the purpose of comparison and because no samples of this cereal were included among those first examined for the present investigation.

A comparison of the analyses reported in this bulletin with those given by the Bureau of Chemistry in previous bulletins and with those reported by König is given in Table XXXIV.

TABLE XXXIV.—*Maize—digestible nutrients averaged by groups.*

[Pounds per hundred pounds of dry matter.]

Character of samples.	Number of samples.	Nutrients.								Production value.		Nutri- tive ratio.
		Protein.		Fat.		Crude fiber.		Carbohydrate.		Gain in flesh.	Calories.	
		Total.	Di- gest- ible.	Total.	Di- gest- ible.	Total.	Di- gest- ible.	Total.	Di- gest- ible.			
Domestic, United States, 1906.....	12	9.90	7.14	4.40	3.92	2.21	1.28	81.96	77.86	23.36	100,965	12.3
Typical, United States, 1898.....		11.21	8.05	4.76	4.28	1.96	.80	80.40	76.38	23.28	100,640	10.8
World's Fair, 1893.....	18	11.09	7.98	4.68	4.21	1.92	.79	80.78	76.74	23.31	100,770	10.9
Jenkins and Winton.....	208	11.78	8.48	6.06	5.45	2.36	.97	78.12	74.20	23.50	101,590	10.3
Foreign, König.....	137	10.91	7.85	4.95	4.45	2.64	1.08	80.01	76.01	23.39	101,115	11.1

From Table XXXIV it is seen that while the domestic maize analyzed in 1906 is considerably lower in protein than the other United States samples and than the foreign, the difference between domestic and foreign grain does not seem to be as marked in this case as with some of the other cereals. In the production value the difference is even less, the extreme variation being only 0.22 pound of flesh gained. The most striking point, however, is that the production value is much higher and the nutritive ratio much broader in the case of maize than for any other cereal.

GENERAL SUMMARY.

Considering, now, the general results in regard to the feeding value of the cereal grains as obtained in this study it appears that, as feeding materials, they fall into three classes typical of the three grains most commonly used, namely, wheat, oats, and maize (or corn). Within one or the other of these groups all of the cereals may be included. This is equally true of those grains commonly grown, such as barley and rye, and of those much less common, such as emmer and proso. Collecting the results as given in the tables of averages (Tables XVIII, XXII, XXIV, XXVI, XXXI, XXXIII) it is seen how distinct this grouping is.

TABLE XXXV.—*Oat group—digestible nutrients.*

[Pounds per hundred pounds of dry matter.]

Cereal.	Protein.	Fat.	Crude fiber.	Carbohydrate.	Production value—gain in flesh.	Nutritive ratio.
Oats.....	10.73	3.59	3.17	51.04	17.86	5.8
Emmer.....	9.96	1.36	4.98	52.06	17.20	6.0
Einkorn.....	11.00	1.55	5.96	48.04	16.81	5.2

TABLE XXXVI.—*Wheat group—digestible nutrients.*

[Pounds per hundred pounds of dry matter.]

Cereal.	Protein.	Fat.	Crude fiber.	Carbo-hydrate.	Production value—gain in flesh.	Nutritive ratio.
Wheat.....	11.93	1.49	1.30	72.42	21.86	6.5
Barley.....	9.37	1.66	1.86	60.96	20.88	8.0
Rye.....	11.29	1.17	1.22	73.82	21.87	6.9

TABLE XXXVII.—*Maize group—digestible nutrients.*

[Pounds per hundred pounds of dry matter.]

Cereal.	Protein	Fat.	Crude fiber.	Carbo-hydrate.	Production value—gain in flesh.	Nutritive ratio.
Maize.....	7.14	3.92	1.28	77.86	23.36	12.3
Proso.....	6.28	2.61	6.11	60.69	19.37	11.5
Sorghum.....	7.61	2.27	.92	74.24	21.62	10.5

OAT GROUP.

In the oat group, as was stated in the introduction, are found those grains which, being high in protein and high in crude fiber, are low in carbohydrate and possess a relatively lower production value and a narrow nutritive ratio. The production value alone can not, however, be taken as a basis for judgment of the value of these grains as feeds.

It is due to the large amount of crude fiber that the amount of digestible carbohydrate is low, thus lowering the production value. At the same time, however, this low carbohydrate makes the relative amount of protein to carbohydrate high, and the grains are characterized as high protein, energy-producing foods rather than heat-producing like those rich in carbohydrate.

The value, therefore, of the oat class of cereals is that they possess high protein and a narrow nutritive ratio. With the less common grains, emmer and einkorn, it is seen that while the protein in emmer is somewhat lower, the production value and nutritive ratio are nearly the same as that of oats. Einkorn, owing to higher crude fiber, falls below the other two in production value and nutritive ratio. It is plain, however, that both of these grains belong in the oat class rather than in the wheat class. While in fat content they resemble wheat and not oats, and in protein content they could be classed with either, as this factor alone is almost the same in the two classes, the large amount of crude fiber, with its resulting effect upon production value and nutritive ratio, places them defi-

nately with the oats. The hulled emmer would, if fed as such, be as much like wheat as it is, unhulled, like oats. As to the intrinsic nutritive value of these three grains of the oat class, our only criterion is the actual production value in Calories of heat energy which 100 pounds of each grain possesses. From this point of view they would rank as follows: Oats, having a value of 17.86 pounds of flesh gained per 100 pounds of grain fed, or 77,209 Calories of heat energy; emmer, with 17.20 pounds of flesh gained, or 74,356 Calories, and einkorn with 16.81 pounds of flesh gained, or 72,670 Calories. These differences in production value are not, however, important, since equal differences are often found between samples of each one of these grains themselves. The average protein content of emmer is nearly 1 per cent lower than that of oats, yet there are 108 samples out of 242 of United States oats which have a protein content less than the average of the emmer, and 7 samples of emmer out of 25 have a higher protein content than the average of the oats. The maximum protein content for emmer was found in the case of No. 1051, with 14.02 pounds of digestible protein, the maximum in the case of the oats being No. 1297, with 15.40 pounds of digestible protein; the minimum protein content for emmer was 6.47 pounds digestible, and for oats 7.12 pounds. For the production value the variations are less marked. Only 21 samples of oats out of 242 fall below 17.20 pounds, the average production value for emmer, while the maximum value for emmer is 17.59 pounds (No. 1061), which is below the average for oats, the maximum production value of einkorn being 17.04 pounds for No. 1068.

While, therefore, there is considerable natural difference between emmer and einkorn on the one hand and oats on the other, this difference seems to be no more marked than similar differences between different samples of oats. For feeding purposes then, the less common cereals, emmer and einkorn, when used without the removal of the hulls, may be considered as belonging with oats in the group of high protein, muscle, or energy-producing foods and nearly equal to the latter in intrinsic food value.

WHEAT GROUP.

In the wheat group of cereals are the three common grains—wheat, barley, and rye. This group is characterized by high protein, as is the oat group, but differs from it in having low crude fiber, which results in a high digestible carbohydrate. The production value is, therefore, high and the nutritive ratio broader than in the oat group, though not broad as compared with other high carbohydrate foods. In this group, therefore, are found foods

which, although high in carbohydrate and in intrinsic food value, yet, because of their high protein, possess an average nutritive ratio and are muscle producing rather than heat producing. On this account wheat has been considered the standard feeding grain where fattening was not the sole object.

When compared with each other and judged by their production values the three members of this group will rank wheat, rye, and barley, the wheat and rye being practically identical in their production value, though the nutritive ratio is narrower and the protein higher in the wheat. These two grains, therefore, are more nearly alike in their composition and value than any other two cereals.

In the case of barley there is a greater difference, due primarily to a considerably lower protein content. This, together with a higher crude fiber, makes the production value lower and the nutritive ratio broader than either of the other two members of this group. Four samples of barley had a protein content higher than the average for wheat, but no barley equaled the wheat average in production value. The effort, however, in this country to grow and breed barleys for high-protein content will probably result in raising the average in this respect and in producing feeding barleys that are more nearly equal to wheat in value.

MAIZE GROUP.

In the last group of cereals—the maize group—are found grains more distinctly different from the other two groups than these are from each other. While the other two groups are similar in being high-protein, energy-producing foods, the maize-group cereals are low in protein. Maize, the typical cereal of the group, is characteristically a high carbohydrate, fat or heat producing food, and because of its relatively low protein possesses a very broad nutritive ratio. The intrinsic food value is higher than that of any of the other cereals. Taken as a group, however, the two distinguishing characteristics are low-protein content and broad nutritive ratio.

The two less common cereals, proso and nonsaccharine sorghum, plainly belong to the maize group when judged by these two standards. In production value both the proso and the sorghum are lower than wheat and rye, and in crude fiber proso is more like the oat group, yet the protein in both is very low and the nutritive ratio much broader than any other cereal except maize. In fat content proso and sorghum are higher than all others except oats and maize. Proso differs from maize and sorghum principally in the amount of crude fiber, which necessarily lowers the amount of digestible carbohydrate. The nutritive ratio, however, is broader than that of sorghum.

Therefore, while proso and sorghum clearly belong with maize in the group of low protein, broad nutritive ratio, fat or heat producing foods, in their intrinsic food value they fall considerably below it. Sorghum in this respect is of about equal value with wheat, while proso is below that of barley, though above oats, maize being higher in its food value than any other cereal.

CONCLUSIONS.

The conclusions drawn from the work presented in this bulletin may be expressed as follows:

(1) The effect of the introduction into the United States of foreign varieties of cereals has been to increase the protein content of the grain. Only two cereals were studied in regard to this point, namely, oats and barley. With both of these grains this increase in protein is an improvement. It increases the high-protein, muscle-producing value of the oats and raises the intrinsic food value of the barley, so that it becomes more nearly equal to that of wheat. Selection and breeding for high-protein content in feeding barleys is a distinct improvement in their value for this purpose.

(2) As feeding grains the cereals may be grouped into three classes typified by our three most common grains, namely, (a) oat group, (b) wheat group, (c) maize group.

Of these three groups the oat group stands at one end as a typical muscle or energy producing food and the maize group at the other end as fat or heat producing. The wheat group is intermediate between these two.

In the oat group belong the less common cereals, emmer and einkorn, and in the maize group proso and nonsaccharine sorghum.

(3) The two cereals emmer (*Triticum dicoccum*) and einkorn (*Triticum monococcum*) belong to the same group as the oats and like it are characterized by high protein and relatively low carbohydrate. They have a lower intrinsic food value than the other grains, but a narrow nutritive ratio and consequently are muscle or energy producers. In food value they are nearly the same as oats, and their cultivation where oats can not be grown is to be advocated.

(4) The two cereals proso or broom-corn millet (*Panicum miliaceum*) and the nonsaccharine sorghum (*Andropogon sorghum*) belong to the maize group, and though lower in food value than maize itself they equal the oat group. They are of great importance when maize can not be grown and when there is need for a high carbohydrate and heat or fat producing food.

(5) Barley and rye belong to the wheat group and are nearly equal to it in food value.

DETAILED TABULATION OF ANALYTICAL AND CALCULATED RESULTS.

TABLE XXXVIII.—Oats—individual analyses.

(Pounds per hundred pounds of dry matter.)

Laboratory No.	Variety.	Grain investigation No.	Locality.	Year and crop.	Water.		Dry matter.		Protein. (N X 6.25.)			Fat.		Crude fiber.		Carbohydrate.		Total production value.		
									Total.	Digestible.	Production value in flesh.	Total.	Digestible.	Production value in flesh.	Total.	Digestible.	Production value in flesh.	Calories.	Nutritive ratio.	
286	Pfiffelbacher's Prolific	295	Germany	Original	9.85	90.15	4.04	12.06	9.41	5.05	2.21	4.19	2.20	10.77	2.80	52.42	13.00	18.10	78,246	6.9
287	Giant Thick Head	296	do.	do.	9.86	90.14	4.27	13.19	10.26	4.90	2.42	4.19	2.14	11.46	2.98	50.95	12.63	17.93	77,511	6.1
288	Gray Winter	299	do.	do.	9.52	90.48	4.25	12.94	10.00	3.77	2.73	5.83	3.07	9.33	2.43	51.15	12.96	18.72	80,926	8.6
289	Pewssammer Hammerich	302	do.	do.	9.03	90.97	3.84	10.00	7.80	1.83	1.83	6.40	5.31	11.77	3.06	52.35	12.92	18.36	79,370	7.4
290	Andarbeck	303	do.	do.	9.34	90.66	3.94	11.44	8.92	2.10	5.77	4.79	2.53	11.18	2.91	72.67	12.96	18.26	78,938	8.5
291	Bavarian Mountain	304	do.	do.	9.18	90.82	3.60	10.06	7.85	1.84	5.78	4.80	2.53	12.77	3.32	82.67	12.96	18.16	78,506	7.4
292	Victoria	305	do.	do.	9.17	90.83	3.67	10.19	7.95	1.87	6.04	5.01	2.64	11.06	2.88	71.69	13.16	18.38	79,457	7.1
293	Longfellow	306	do.	do.	9.80	90.30	3.97	11.87	9.26	2.18	6.04	5.01	2.64	11.41	2.97	74.66	12.74	18.30	79,111	8.5
294	Danish Island	308	do.	do.	9.67	90.33	3.87	11.50	8.97	2.11	5.59	4.64	2.44	11.98	2.85	71.68	12.96	18.25	78,805	7.3
295	Yellow Thuringen.	309	do.	do.	9.87	91.03	4.70	11.19	8.73	2.05	6.14	5.10	2.68	10.64	2.77	65.69	13.16	18.28	79,024	7.6
296	Milton	310	do.	do.	9.14	90.86	4.01	11.44	8.92	2.05	5.37	4.46	2.35	10.55	2.94	67.67	13.16	18.31	79,154	8.5
297	Waterloo.	312	do.	do.	9.23	90.77	3.81	10.06	7.85	1.84	5.68	4.71	2.48	10.86	2.82	69.67	13.24	18.30	79,243	7.4
298	Yellow Giant.	314	do.	do.	9.19	90.81	4.12	11.44	8.92	2.05	6.14	5.10	2.68	10.64	2.77	65.69	13.16	18.30	79,154	8.5
299	Yellow Flanders.	316	do.	do.	9.19	90.81	4.12	11.44	8.92	2.05	6.14	5.10	2.68	10.64	2.77	65.69	13.16	18.30	79,154	8.5
300	Georgian	317	do.	do.	8.93	91.07	4.06	10.75	8.38	1.97	6.55	5.44	3.02	10.48	2.72	67.66	12.84	18.59	80,364	7.1
301	White Polish.	318	do.	do.	10.30	89.70	4.40	10.94	8.53	2.22	6.55	5.44	3.02	10.48	2.72	67.66	12.84	18.20	78,679	8.0
302	Rugen.	319	do.	do.	10.34	89.86	4.34	11.87	9.26	2.18	6.04	5.01	2.64	11.41	2.97	74.66	12.96	18.30	79,111	7.6
303	Barlachov	321	do.	do.	10.51	89.49	4.32	10.94	8.53	2.18	6.04	5.01	2.64	11.41	2.97	74.66	12.96	18.30	79,111	7.6
304	Polodia.	322	do.	do.	10.51	89.49	4.32	10.94	8.53	2.18	6.04	5.01	2.64	11.41	2.97	74.66	12.96	18.30	79,111	7.6
305	White Swedish.	323	do.	do.	9.68	90.32	3.94	9.68	7.56	1.78	5.64	4.68	2.46	11.36	2.69	67.66	12.84	18.48	79,243	7.4
306	England.	288	England.	do.	7.50	92.50	3.33	12.00	9.36	2.20	5.64	4.98	2.46	11.38	2.94	73.68	13.24	18.21	78,549	6.9
307	Polodia.	289	do.	do.	7.35	90.65	3.19	11.87	9.26	2.18	5.68	4.72	2.48	11.76	3.00	67.67	13.24	18.17	78,549	6.9
308	Polodia.	290	do.	do.	7.29	92.71	3.15	11.87	9.26	2.18	5.68	4.72	2.48	11.76	3.00	67.67	13.24	18.17	78,549	6.9
309	Polodia.	291	do.	do.	11.40	88.60	3.02	12.19	9.51	2.23	7.31	6.07	3.13	8.38	2.18	67.67	13.24	18.30	79,243	7.4
310	Polodia.	292	do.	do.	6.89	93.11	2.78	9.94	7.75	1.82	4.94	4.10	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
311	Polodia.	293	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
312	Polodia.	294	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
313	Polodia.	295	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
314	Polodia.	296	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
315	Polodia.	297	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
316	Polodia.	298	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
317	Polodia.	299	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
318	Polodia.	300	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
319	Polodia.	301	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
320	Polodia.	302	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
321	Polodia.	303	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
322	Polodia.	304	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
323	Polodia.	305	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
324	Polodia.	306	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
325	Polodia.	307	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
326	Polodia.	308	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
327	Polodia.	309	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
328	Polodia.	310	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
329	Polodia.	311	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
330	Polodia.	312	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
331	Polodia.	313	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
332	Polodia.	314	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
333	Polodia.	315	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
334	Polodia.	316	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
335	Polodia.	317	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
336	Polodia.	318	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
337	Polodia.	319	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
338	Polodia.	320	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
339	Polodia.	321	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
340	Polodia.	322	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
341	Polodia.	323	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
342	Polodia.	324	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
343	Polodia.	325	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4
344	Polodia.	326	do.	do.	7.12	92.88	2.68	10.56	8.10	1.94	4.86	4.04	2.16	11.22	2.92	72.71	13.24	18.30	79,243	7.4

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TABLE XXXVIII.—Oats—Individual analyses—Continued.

Laboratory No.	Variety.	Grain investigation No.	Locality.	Year and crop.	Protein (N X 6.25).			Fat.			Crude fiber.		Carbohydrate.			Total production value.		Nutritive ratio.
					Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Gain in flesh.	
1267	Swedish Select.	213	N. C.	1905 (3)	11.86	9.27	2.18	3.85	2.81	1.24	12.40	3.22	0.80	69.04	13.18	17.40	75.220	6.9
1268	do.	213	Ind.	1903 (1)	12.69	9.90	2.33	3.39	2.81	1.48	12.06	3.14	0.78	68.47	13.07	17.66	76.344	6.3
1269	do.	213	do.	1904 (1)	13.38	10.43	2.45	3.87	2.67	1.23	10.96	2.85	0.75	68.28	13.04	17.64	76.258	5.9
1270	do.	213	do.	1905 (1)	14.19	11.07	2.60	4.57	2.38	1.09	10.96	2.85	0.74	68.11	13.06	17.56	75.912	5.5
1271	Idaho	213	Idaho	1904	10.66	8.34	1.96	4.57	3.09	2.13	9.98	2.59	0.64	67.16	13.06	17.79	76.906	7.7
1272	do.	213	do.	1905 (1)	13.31	10.36	2.44	4.37	2.04	1.03	9.87	2.58	0.64	67.16	13.06	17.79	76.944	6.1
1273	do.	213	do.	1905 (1)	11.50	8.97	2.11	4.09	4.44	2.22	10.56	2.75	0.88	68.12	13.06	17.79	76.944	7.3
1274	Tex.	213	Tex.	1904	14.44	11.26	2.65	4.35	3.29	1.08	10.56	2.75	0.88	68.12	13.06	17.79	76.944	7.3
1275	do.	213	do.	1905 (2)	15.31	11.94	2.81	4.22	3.54	1.14	10.56	2.75	0.88	68.12	13.06	17.79	76.944	7.3
1276	do.	213	Mo.	1903 (1)	11.69	9.31	2.19	3.56	2.94	1.03	10.56	2.75	0.88	68.12	13.06	17.79	76.944	7.3
1277	do.	213	do.	1905 (1)	12.00	9.36	2.24	4.59	1.48	0.78	10.56	2.75	0.88	68.12	13.06	17.79	76.944	7.3
1278	N. Dak.	213	N. Dak.	1905 (1)	12.94	10.09	2.37	3.62	2.40	1.08	10.56	2.75	0.88	68.12	13.06	17.79	76.944	7.3
1279	do.	213	do.	1905 (3)	12.94	10.09	2.37	3.62	2.40	1.08	10.56	2.75	0.88	68.12	13.06	17.79	76.944	7.3
1280	do.	213	Ohio	1905 (1)	13.50	10.53	2.47	4.56	3.91	1.03	10.56	2.75	0.88	68.12	13.06	17.79	76.944	7.3
1281	Wyo.	134	Wyo.	1905 (4)	12.81	9.99	2.35	3.48	3.07	1.03	10.56	2.75	0.88	68.12	13.06	17.79	76.944	7.3
1282	Me.	213	Me.	1905 (4)	13.81	10.72	2.52	4.89	3.25	1.99	11.02	2.86	0.71	67.70	13.21	18.22	77.197	5.9
1283	do.	213	Canada	1905 (2)	13.75	10.72	2.52	4.89	3.25	1.99	11.02	2.86	0.71	67.70	13.21	18.22	77.197	5.9
1284	do.	213	do.	1905 (2)	13.81	10.72	2.52	4.89	3.25	1.99	11.02	2.86	0.71	67.70	13.21	18.22	77.197	5.9
1285	Kans.	165	Kans.	1905 (1)	13.87	10.82	2.54	5.19	3.79	1.30	11.47	2.98	0.66	68.06	13.15	18.26	78.024	5.5
1286	Ne-br.	165	Ne-br.	1905 (1)	15.19	11.85	2.78	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1287	do.	165	N. Dak.	1903	15.44	12.04	2.83	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1288	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1289	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1290	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1291	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1292	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1293	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1294	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1295	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1296	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1297	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1298	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1299	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1300	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1301	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1302	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1303	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1304	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1305	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1306	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1307	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1308	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1309	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1310	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1311	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1312	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1313	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1314	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1315	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1316	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1317	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1318	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1319	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1320	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1321	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1322	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1323	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0
1324	do.	165	do.	1905 (1)	15.56	12.14	2.85	6.22	4.97	1.94	10.88	2.83	0.70	66.91	13.07	17.52	74.356	5.0

1285	do.	165	1002 (1)	12.43	2.96	1.57	10.82	2.81	70.60	4.8	76.600	4.8
1286	do.	165	1004 (3)	15.04	3.67	2.11	10.22	2.66	78.240	4.7	78.240	4.7
1287	Wash.	165	1005 (2)	12.51	4.09	2.40	9.06	2.36	78.765	4.7	78.765	4.7
1288	Tex.	165	1006 (1)	16.81	4.14	2.49	12.03	2.96	75.470	4.8	75.470	4.8
1289	Idaho	165	1005 (1)	15.75	4.59	1.60	10.85	2.64	76.903	7.4	76.903	7.4
1290	Wyo.	165	1005 (1)	11.06	8.63	1.94	8.85	2.34	78.906	5.9	78.906	5.9
1291	do.	165	1005 (1)	13.87	8.63	1.60	10.85	2.34	75.600	6.0	75.600	6.0
1292	do.	165	1005 (1)	12.04	4.70	1.66	13.67	2.62	75.307	8.9	75.307	8.9
1293	do.	165	1005 (1)	16.25	10.07	1.75	11.28	2.55	76.983	5.5	76.983	5.5
1294	Cal.	165	1005 (1)	9.19	4.41	1.68	10.27	2.67	74.053	4.9	74.053	4.9
1295	Oreg.	165	1005 (1)	14.44	4.31	1.49	12.52	2.36	76.820	4.1	76.820	4.1
1296	do.	165	1005 (1)	15.25	5.44	1.63	10.92	2.64	76.387	3.7	76.387	3.7
1297	Utah	165	1005 (1)	18.19	11.98	1.49	11.67	2.03	76.258	6.0	76.258	6.0
1298	Ohio	165	1005 (1)	19.75	15.40	1.49	11.85	2.03	76.338	6.3	76.338	6.3
1299	do.	165	1005 (1)	13.13	9.99	1.66	11.23	2.92	79.111	6.0	79.111	6.0
1300	N.Y.	165	1005 (1)	12.81	9.99	1.66	11.23	2.92	77.338	6.0	77.338	6.0
1301	Ill.	165	1005 (1)	14.44	12.82	1.54	9.45	2.27	76.781	6.1	76.781	6.1
1302	Minn.	165	1005 (1)	13.44	10.48	1.54	11.25	2.46	75.470	4.9	75.470	4.9
1303	S.C.	165	1005 (1)	12.04	4.70	1.31	12.05	2.33	76.820	4.1	76.820	4.1
1304	Mo.	165	1004 (2)	13.04	10.14	1.31	12.05	2.33	77.468	5.2	77.468	5.2
1305	do.	165	1005 (3)	12.69	12.53	2.02	11.45	3.10	74.917	7.8	74.917	7.8
1311	Tobolsk.	135	1003 (3)	16.06	12.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1312	do.	135	1003 (3)	18.06	16.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1313	N. Dak.	135	1002 (3)	12.63	10.63	2.10	12.13	3.45	76.387	3.7	76.387	3.7
1314	S. Dak.	135	1004 (3)	14.04	11.64	2.31	14.54	3.78	77.814	5.8	77.814	5.8
1315	do.	135	1004 (3)	10.44	8.94	2.31	14.54	3.78	77.338	6.2	77.338	6.2
1316	do.	135	1002 (3)	12.75	11.11	2.31	14.54	3.78	76.820	4.1	76.820	4.1
1317	do.	135	1004 (3)	14.25	11.11	2.31	14.54	3.78	77.814	5.8	77.814	5.8
1318	do.	135	1004 (3)	11.25	11.11	2.31	14.54	3.78	77.338	6.2	77.338	6.2
1319	do.	135	1004 (3)	13.13	11.11	2.31	14.54	3.78	76.820	4.1	76.820	4.1
1320	Shatlov.	144	1004 (3)	10.77	7.60	2.50	15.43	4.01	77.814	5.8	77.814	5.8
1321	do.	144	1005 (3)	13.31	10.77	2.50	15.43	4.01	77.338	6.2	77.338	6.2
1322	Belgian Winter.	166	1003 (3)	13.31	10.77	2.50	15.43	4.01	76.820	4.1	76.820	4.1
1323	do.	166	1003 (3)	13.31	10.77	2.50	15.43	4.01	77.814	5.8	77.814	5.8
1324	do.	166	1003 (3)	13.31	10.77	2.50	15.43	4.01	77.338	6.2	77.338	6.2
1325	do.	166	1003 (3)	13.31	10.77	2.50	15.43	4.01	76.820	4.1	76.820	4.1
1326	North Finnish Black.	278	1004 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1327	do.	167	1004 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1328	do.	174	1004 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1329	do.	174	1004 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1330	do.	174	1004 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1331	do.	174	1004 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1332	do.	174	1004 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1333	do.	174	1004 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1334	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1335	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1336	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1337	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1338	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1339	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1340	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1341	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1342	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1343	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1344	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1345	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1346	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1347	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1348	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1349	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1350	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1351	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1352	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1353	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1354	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1355	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1356	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1357	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1358	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1359	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1360	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1361	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1362	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1363	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1364	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1365	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1366	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1367	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1368	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1369	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1370	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1371	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1372	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1373	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1374	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1375	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1376	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1377	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1378	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1379	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1380	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1381	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1382	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1383	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1384	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.338	6.2	77.338	6.2
1385	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	76.820	4.1	76.820	4.1
1386	do.	283	1003 (3)	12.63	10.63	2.10	12.13	3.45	77.814	5.8	77.814	5.8
1387	do.	283	1003 (3)	12.63	10.63							

TABLE XXXVIII.—Oats—individual analyses—Continued.

Laboratory No.	Variety.	Locality.	Year and crop.	Grain investigation No.	Water.		Dry matter.	Protein. (N X 6.25.)			Fat.			Crude fiber.		Carbohydrate.			Total production value.		Nutritive ratio.	
								Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Gain in flesh.		Calories.
339	Khorson.	Nebr.	1904	212	8.58	91.42	3.93	12.13	9.46	2.22	3.30	1.74	12.67	29.73	0	0.28	15.15	67.29	51.15	10.37	65,493	6.6
340	Red Rust Proof.	Kans.	1905 (3)	256	8.19	91.81	3.43	14.44	11.26	2.55	4.42	2.33	10.38	22.94	73	73	18.33	66.42	51.14	12.38	79,240	5.7
348	Texas Red Rust Proof.	do.	1905 (1)	451	7.02	92.98	3.75	13.44	10.48	2.46	4.96	2.17	11.29	32.94	73	73	18.03	64.82	48.92	12.38	77,944	5.6
358	do.	Kans.	1904 (1)	458	7.72	92.28	4.60	10.31	8.04	1.89	4.58	2.36	12.66	32.94	73	73	17.68	64.93	48.76	12.34	77,944	5.6
369	do.	Tex.	1905 (2)	458	7.86	92.14	4.07	10.31	8.04	1.89	4.58	2.36	14.18	33.99	92	92	18.31	65.51	50.44	12.51	76,541	7.4
370	do.	do.	1904 (1)	458	7.69	92.31	3.15	15.19	11.53	2.78	4.74	2.49	11.31	32.94	92	92	18.17	66.34	48.36	12.24	76,541	7.4
371	Apple's Rust Proof.	N. Cal.	1905 (1)	320	8.19	91.82	3.96	12.38	9.66	2.62	4.74	2.62	11.58	33.99	92	92	18.31	65.51	50.44	12.51	79,154	5.8
384	Culbertson Winter.	Cal.	1905 (1)	339	8.94	93.06	3.85	11.00	8.58	2.02	5.45	2.81	11.26	22.93	65	65	18.09	67.71	52.14	12.64	79,457	7.9
376	do.	Md.	1906	273	8.84	91.16	2.82	15.37	11.99	2.02	6.54	2.86	10.64	22.71	69	69	18.70	64.90	49.44	12.33	80,840	5.4
332	do.	Tex.	1904 (1)	273	7.67	92.33	4.15	12.75	9.94	1.92	5.46	2.35	12.51	33.51	87	87	17.89	67.15	51.28	12.52	78,203	6.4
342	do.	Ill.	1903 (1)	273	8.07	91.33	3.94	9.94	7.75	1.92	5.46	2.35	13.51	35.1	87	87	18.09	66.00	50.73	12.58	78,203	6.4
372	do.	Md.	1904 (1)	273	9.22	90.78	3.02	13.04	10.19	2.30	6.75	2.60	11.39	22.96	73	73	18.37	65.88	50.73	12.52	79,716	6.5
373	do.	do.	1905 (2)	273	9.22	90.78	3.02	13.04	10.19	2.30	6.75	2.60	11.39	22.96	73	73	18.37	65.88	50.73	12.52	79,716	6.5
374	do.	Kans.	1905 (2)	219	8.66	91.34	4.05	17.38	13.56	3.19	6.23	2.77	11.81	33.07	76	76	19.96	64.12	47.92	11.88	77,427	5.9
346	Prince Edward's Island.	do.	1905 (3)	286	8.04	91.36	4.47	13.69	10.08	2.75	5.75	2.72	10.11	22.63	55	55	18.37	64.12	47.92	11.88	77,427	5.9
347	do.	N. Dak.	1905 (2)	286	8.09	90.91	4.08	15.06	11.75	2.76	5.82	2.84	12.19	33.07	76	76	19.96	64.12	47.92	11.88	77,427	5.9
348	do.	S. Dak.	1905 (2)	286	8.09	90.91	3.94	12.19	9.51	2.76	5.82	2.84	12.19	33.07	76	76	19.96	64.12	47.92	11.88	77,427	5.9
349	do.	do.	1905 (2)	286	8.65	91.14	4.07	13.37	10.43	2.45	4.85	2.12	11.78	33.06	92	92	17.09	64.62	49.76	12.34	78,851	6.6
350	do.	Tex.	1905 (1)	286	8.65	91.35	4.05	13.37	10.43	2.61	4.85	2.12	11.78	33.06	92	92	17.09	64.62	49.76	12.34	78,851	6.6
351	do.	do.	1905 (1)	286	8.76	91.24	5.27	11.25	8.77	2.07	4.56	3.75	12.99	33.83	84	84	17.80	65.33	50.30	12.47	76,949	6.6
352	do.	Oreg.	1905 (1)	286	8.55	91.45	6.25	16.19	12.93	2.97	4.77	3.96	9.52	32.48	61	61	17.46	65.65	50.55	12.54	76,431	5.5
353	do.	Utah.	1905 (1)	286	8.79	92.01	5.34	13.81	10.77	2.15	5.01	4.16	12.92	33.36	83	83	17.56	65.92	50.55	12.08	75,912	5.7
354	do.	Ariz.	1905 (1)	286	8.29	91.71	4.91	11.75	9.16	2.15	5.01	4.16	13.17	33.42	85	85	17.70	64.87	49.95	12.39	76,517	6.9
379	Unnamed varieties.	Wash.	1905 (2)	288	8.07	91.33	4.02	10.31	8.04	1.89	6.38	3.07	11.93	33.10	77	77	18.32	67.97	50.64	12.34	79,197	8.3
335	do.	do.	1905 (2)	288	7.80	92.20	4.82	10.31	8.53	1.89	6.38	3.07	13.73	33.57	89	89	17.79	65.57	50.64	12.98	77,165	6.3
336	do.	do.	1905 (2)	288	8.41	91.59	4.43	13.56	10.58	2.45	4.87	5.16	10.02	22.61	73	73	18.32	67.98	52.14	12.68	77,917	6.9
337	do.	do.	1905 (2)	288	8.24	91.76	3.71	12.81	9.99	2.39	4.41	3.41	11.39	22.96	94	94	18.24	66.40	50.28	12.47	75,307	6.1
338	do.	do.	1905 (2)	291	8.02	91.98	3.89	11.44	8.92	2.07	3.70	3.07	13.92	33.62	75	75	18.41	66.40	50.28	12.47	75,307	6.1
339	do.	Cal.	1905 (1)	293	8.54	91.46	4.00	12.94	10.09	2.37	6.95	5.77	11.70	33.04	90	90	18.24	66.40	50.28	12.47	75,307	6.1
340	do.	Kans.	1905 (2)	293	8.81	91.19	5.10	14.00	10.92	2.57	6.95	5.77	11.70	33.04	90	90	18.24	66.40	50.28	12.47	75,307	6.1
361	Burt.	do.	1904 (1)	283	8.26	91.74	5.32	12.81	9.99	2.38	6.95	5.45	12.80	22.96	73	73	18.43	67.98	52.14	12.03	78,549	6.4
362	do.	N. Dak.	1904 (1)	283	8.93	91.62	4.59	15.69	12.54	2.89	6.95	5.45	12.80	22.96	73	73	18.43	67.98	52.14	12.03	78,549	6.4
363	do.	Tex.	1905 (2)	293	7.93	92.07	4.45	13.56	10.58	2.48	6.95	5.45	12.80	22.96	73	73	18.43	67.98	52.14	12.03	78,549	6.4
364	do.	do.	1905 (2)	293	7.95	92.05	4.20	15.19	11.85	2.47	6.12	5.08	12.67	31.17	79	79	18.22	68.00	46.84	12.12	77,857	6.0
365	do.	do.	1904 (1)	293	9.08	90.92	3.81	13.50	10.63	2.81	6.90	4.65	11.23	22.92	72	72	18.22	68.00	46.84	12.12	77,857	6.0

TABLE XXXIX.—*Emmer—individual analyses.*

[Founds per hundred pounds of dry matter.]

TABLE XXXIX.— <i>Emmer—individual analyses.</i>																									
[Pounds per hundred pounds of dry matter.]																									
Laboratory No.	Variety.	Grain investigation No.	Locality.	Year and crop.	Water.	Dry matter.	Protein (N×6.25).			Fat.			Crude fiber.			Carbohydrate.			Total production value.		Nutritive ratio.				
							Total.	Digestible.	Production value—in flesh.	Total.	Digestible.	Production value—in flesh.	Total.	Digestible.	Production value—in flesh.	Total.	Digestible.	Production value—in flesh.	Calories.						
293	do.	293	do.	1905 (2)	9.49	90.51	3.14	15.63	12.19	2.86	5.63	4.67	2.46	10.55	2.75	68	65.04	50.08	12.42	18.47	70.630	5.2			
294	Balyak.	294	Cal.	1905 (1)	8.42	91.58	3.16	12.63	9.85	2.31	5.26	3.55	1.87	13.84	3.60	80	65.08	50.12	12.43	17.50	70.586	6.3			
295	Snoma.	295	Cal.	1905 (1)	9.25	90.75	3.94	12.31	9.60	2.36	5.31	3.55	1.87	13.84	3.60	80	65.08	50.12	12.43	17.50	70.586	6.3			
296	do.	296	Cal.	1905 (1)	8.45	91.55	2.94	14.21	11.07	2.06	5.31	3.55	1.87	13.84	3.60	80	65.08	50.12	12.43	17.50	70.586	6.3			
297	Big Four.	297	Ill.	1905 (1)	6.75	93.25	3.73	11.25	8.77	2.06	5.31	3.55	1.87	13.84	3.60	80	65.08	50.12	12.43	17.50	70.586	6.3			
298	White Russian.	298	N. Dak.	1902	6.32	93.68	3.49	12.44	10.57	2.55	5.03	4.03	2.45	15.60	2.68	100	65.85	50.70	12.52	18.44	70.718	7.2			
298	Black Hungarian.	298	N. Mex.	1902	9.12	90.88	5.37	12.44	9.70	2.98	5.03	4.03	2.45	15.60	2.68	100	65.85	50.70	12.52	18.44	70.718	7.2			
298	Ligowo.	298	Minn.	1902	10.05	89.95	3.27	11.12	8.67	2.04	5.31	4.32	2.27	10.44	2.71	67	64.90	50.02	12.90	17.38	75.134	7.2			

TABLE XL.—*Einkorn* — individual analyses.

[Pounds per hundred pounds of dry matter.]

Laboratory No.	Grain investigation No.	Locality.	Year and crop.	Water.	Dry Matter.	Ash.	Protein (N X 6.25).				Fat.				Crude fiber.			Carbohydrate.				Total production value.		Nutritive ratio.
							Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Gain in flesh.	Calories.	
065	2433	Kans.	1905 (1)	7.86	92.14	6.82	16.25	12.19	2.86	2.14	1.52	0.80	13.15	5.79	1.44	61.64	46.23	11.46	16.56	71,589	4.5			
066	2433	S. Dak.	1905 (2)	8.78	91.22	5.69	13.75	10.31	2.42	2.18	1.56	.82	14.67	6.45	1.60	63.71	47.78	11.85	16.69	72,451	5.6			
067	2433	do	1905 (2)	8.81	91.19	5.04	15.00	11.25	2.64	2.00	1.42	.75	13.39	5.89	1.46	64.57	48.43	12.01	16.86	72,886	5.1			
068	2433	Tex.	1905 (1)	7.91	92.09	4.72	13.69	10.27	2.41	2.43	1.73	.91	13.01	5.72	1.42	66.15	49.61	12.30	17.04	73,664	5.8			

TABLE XLI.—*Wheat* — individual analyses.

[Pounds per hundred pounds of dry matter.]

Laboratory No.	Variety.	Locality.	Water.	Dry matter.	Ash.	Protein (N X 5.7)			Fat.			Crude fiber.			Carbohydrate.			Total production value.			Nutritive ratio.
						Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Gain in flesh.	Calories.		
	Durum.....	Russia.....	11.02	88.98	1.78	16.87	14.17	3.33	2.25	1.42	0.75	2.40	1.13	0.28	76.56	70.50	17.50	21.86	94,501	5.3	
	<i>T. vulgare</i>	do.....	9.43	90.57	1.85	16.76	14.07	3.31	1.72	1.08	.57	2.45	1.15	.28	77.22	71.04	17.62	21.76	94,068	5.3	
	<i>T. sativum</i>	India.....	9.58	90.42	2.30	11.97	10.05	2.36	2.62	1.65	.87	3.06	1.44	.36	80.05	73.65	18.26	21.85	94,457	5.8	
	Durum.....	United States.....	9.77	90.23	2.02	15.50	13.02	3.06	2.73	1.72	.91	2.83	1.33	.33	76.92	70.77	17.55	21.85	94,717	6.6	
	N. W. spring.....	do.....	10.11	89.96	1.85	14.02	11.78	2.77	2.24	1.41	.74	2.59	1.17	.29	79.39	73.04	18.11	21.91	94,717	6.6	
	Kans. winter.....	do.....	10.68	89.32	2.28	13.17	11.06	2.60	1.81	1.14	.60	2.79	1.31	.32	79.95	73.55	18.24	21.76	94,068	7.0	
	Soft winter.....	do.....	10.55	89.45	2.00	11.86	9.96	2.34	1.91	1.20	.63	2.49	1.17	.29	81.77	75.24	18.66	21.92	94,760	7.9	

TABLE XLII.—*Rye — individual analyses.*

[Pounds per hundred pounds of dry matter.]

Laboratory No.	Variety.	Grain investigation No.	Locality.	Year and crop.	Water.	Dry matter.	Ash.	Protein (N X 6.25).			Fat.			Crude fiber.			Carbohydrate.			Total production value.		Nutritive ratio.
								Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Gain in flesh.	Calories.	
1069	Ivanov.....	30	Md.....	1906 (1)	9.37	90.93	2.27	13.06	10.97	2.58	1.64	1.18	0.82	2.47	1.31	0.32	80.36	73.93	18.33	21.55	94,457	7.1
1070	do.....	34	Kans.....	1905 (1)	9.92	90.08	2.36	17.44	14.65	3.44	1.75	1.12	.59	2.44	1.29	.32	76.01	69.92	17.34	21.69	93,766	7.0
1071	do.....	34	do.....	1905 (1)	9.49	90.51	2.28	13.75	11.55	2.71	1.87	1.20	.63	2.66	1.41	.35	79.44	73.09	18.13	21.82	94,228	6.7
1072	do.....	34	do.....	1905 (1)	9.14	90.86	2.23	11.12	9.34	2.19	1.93	1.24	.65	2.48	1.31	.32	82.24	75.66	18.76	21.92	94,760	8.5
1073	do.....	34	do.....	1905 (1)	9.75	90.25	2.33	12.62	10.59	2.49	2.17	1.39	.73	2.38	1.26	.31	80.50	74.06	18.37	21.90	94,674	7.4
1074	do.....	34	Tex.....	1905 (1)	8.18	90.82	1.65	11.87	9.97	2.34	1.78	1.14	.60	2.23	1.18	.29	82.47	75.87	18.81	22.04	93,279	8.0
1075	do.....	34	Wyo.....	1905 (1)	8.87	91.13	2.08	10.50	8.82	2.07	1.64	.99	.52	2.17	1.15	.28	83.71	77.01	19.10	21.79	94,976	9.1
1076	Abbruzzes.....	40	Kans.....	1905 (1)	9.42	90.58	2.27	15.62	13.11	3.06	1.68	1.08	.57	2.29	1.21	.30	78.14	71.89	17.10	21.78	94,155	5.8
1077	do.....	40	Cal.....	1905 (1)	9.26	90.74	2.09	13.31	11.18	2.63	1.77	1.13	.59	2.15	1.14	.28	80.68	74.22	18.41	22.01	94,717	7.0
1078	do.....	40	do.....	1905 (1)	9.09	90.91	2.09	10.75	9.03	2.12	1.96	1.17	.66	2.16	1.10	.28	83.02	76.38	18.04	22.01	95,149	8.9
1079	do.....	40	Tex.....	1905 (1)	9.13	90.87	1.65	15.44	12.97	3.05	1.77	1.13	.59	2.07	1.10	.33	80.27	73.85	18.32	21.95	94,890	7.6
1080	Spring.....	40	Oreg.....	1904 (1)	8.88	91.12	3.06	12.19	10.24	2.41	1.95	1.25	.66	2.53	1.34	.31	82.19	75.61	18.75	21.72	93,895	8.3
1081	do.....	73	Nebr.....	1905 (2)	9.87	90.13	2.12	11.37	9.55	2.24	1.91	1.24	.64	2.39	1.27	.31	82.19	73.37	18.19	21.92	94,890	7.1
1082	do.....	73	do.....	1905 (1)	9.42	90.58	1.95	14.19	11.92	2.80	1.91	1.09	.57	2.20	1.17	.29	79.75	73.37	18.19	21.75	94,760	5.4
1083	do.....	73	do.....	1905 (1)	9.55	90.45	2.32	16.44	13.81	3.25	1.71	1.06	.57	2.22	1.18	.29	77.31	71.12	17.64	21.84	94,414	6.0
1084	do.....	73	do.....	1905 (1)	9.78	90.22	2.17	15.19	12.76	3.00	1.84	1.18	.62	2.17	1.15	.28	78.63	73.87	18.32	21.88	94,587	9.7

TABLE XLIII.—*Barley — individual analyses.*

[Pounds per hundred pounds of dry matter.]

Laboratory No.	Variety.	Grain Investigation No.	Locality.	Year and crop.	Dry matter.			Protein (N X 6.25).			Fat.			Crude fiber.			Carbohydrate.			Total production value.		Nutritive ratio.
					Water.	Ash.	Total.	Digestible.	Production in flesh—gain	Total.	Digestible.	Production in flesh.	Total.	Digestible.	Production in flesh.	Total.	Digestible.	Production in flesh.	Calories.			
124	Hanna	226	Ariz.	1905 (1)	8.94	91.06	2.55	11.06	7.74	1.82	1.80	1.80	1.45	79.90	73.51	18.23	21.39	92.469	10.2			
225	do.	267	Kans.	1905 (2)	9.63	90.37	2.90	15.44	10.81	2.54	1.90	1.90	1.47	75.11	69.12	17.14	21.02	90.869	9.9			
226	do.	301	do.	1905 (2)	9.54	90.46	2.35	14.31	10.02	2.35	1.90	1.46	1.46	75.79	69.72	17.29	21.00	90.783	7.6			
227	do.	303	do.	1905 (2)	9.25	90.75	3.35	16.00	11.20	2.63	1.90	1.53	1.53	73.87	67.96	16.85	20.80	90.178	8.4			
228	do.	318	do.	1905 (2)	9.59	90.41	3.14	14.75	10.32	2.43	1.74	1.97	1.30	70.10	67.36	17.36	21.09	91.172	7.3			
229	Tennessee winter	257	do.	1905 (2)	9.59	90.43	3.56	13.12	9.18	2.16	1.83	1.95	1.95	75.44	69.40	17.21	20.78	90.832	8.2			
230	do.	257	do.	1905 (2)	9.18	90.82	3.63	12.75	8.92	2.10	1.82	1.90	1.90	76.52	69.00	17.26	20.80	90.178	8.5			
231	do.	257	do.	1905 (2)	9.12	90.88	3.32	12.75	8.92	2.11	1.82	1.94	1.94	76.78	69.39	17.46	20.91	90.394	8.9			
232	do.	257	do.	1905 (2)	8.73	91.37	3.27	12.81	8.97	2.21	1.83	1.97	1.72	76.78	69.39	17.46	20.91	90.394	8.9			
233	do.	257	do.	1905 (2)	8.65	90.35	3.23	14.06	9.83	2.31	1.83	1.98	1.98	76.78	69.39	17.46	20.91	90.394	8.9			
234	do.	257	do.	1905 (2)	10.00	90.00	3.47	11.37	7.96	1.87	1.84	1.84	1.81	76.78	69.39	17.46	20.91	90.394	8.9			
235	do.	257	do.	1905 (2)	8.82	91.18	3.42	11.75	8.22	1.93	1.80	1.78	1.94	76.78	69.39	17.46	20.91	90.394	8.9			
236	do.	257	do.	1905 (2)	8.92	91.18	3.24	12.62	8.83	2.07	1.80	1.98	1.98	76.78	69.39	17.46	20.91	90.394	8.9			
237	do.	257	do.	1905 (2)	9.19	90.81	2.93	14.44	10.11	2.38	1.91	1.70	1.89	76.78	69.39	17.46	20.91	90.394	8.9			
238	do.	257	do.	1905 (2)	9.10	90.90	3.37	13.62	9.53	2.24	1.91	1.55	1.81	76.78	69.39	17.46	20.91	90.394	8.9			
239	do.	257	do.	1905 (2)	9.18	90.82	3.37	12.25	8.57	2.01	1.91	1.70	1.89	76.78	69.39	17.46	20.91	90.394	8.9			
240	do.	257	do.	1905 (2)	9.16	90.84	3.30	12.87	9.01	2.12	1.90	1.78	1.94	76.78	69.39	17.46	20.91	90.394	8.9			
241	do.	257	do.	1905 (2)	8.87	91.13	3.01	15.50	10.50	2.47	1.79	1.59	1.84	76.78	69.39	17.46	20.91	90.394	8.9			
242	do.	257	do.	1905 (1)	9.19	90.81	3.08	11.31	7.92	1.86	1.76	1.93	1.89	76.78	69.39	17.46	20.91	90.394	8.9			
243	do.	257	Tex.	1905 (1)	9.17	90.83	2.71	16.75	11.72	2.05	1.78	1.94	1.89	76.78	69.39	17.46	20.91	90.394	8.9			
244	do.	257	Ind. Ter.	1905 (1)	12.37	87.63	2.33	12.19	8.53	1.73	1.78	1.89	1.89	76.78	69.39	17.46	20.91	90.394	8.9			
245	do.	257	Ind.	1904 (1)	13.28	86.72	3.07	10.50	7.35	2.00	1.59	1.89	1.89	76.78	69.39	17.46	20.91	90.394	8.9			
246	do.	257	Mo.	1905 (2)	9.36	90.64	2.52	12.31	8.62	2.03	1.79	1.91	1.91	76.78	69.39	17.46	20.91	90.394	8.9			
247	do.	257	Kans.	1905 (1)	9.02	90.94	3.28	11.87	8.31	1.95	1.69	1.99	1.99	76.78	69.39	17.46	20.91	90.394	8.9			
248	do.	353	do.	1905 (1)	8.97	91.03	3.10	9.56	6.69	1.57	1.71	1.52	1.99	76.78	69.39	17.46	20.91	90.394	8.9			
249	do.	353	do.	1905 (1)	9.07	91.03	3.07	14.50	10.15	2.39	1.71	1.52	1.99	76.78	69.39	17.46	20.91	90.394	8.9			
250	do.	353	do.	1905 (1)	9.07	91.03	3.23	14.56	10.19	2.40	1.68	1.50	1.99	76.78	69.39	17.46	20.91	90.394	8.9			
251	do.	353	do.	1905 (1)	9.41	90.59	3.04	12.37	8.66	2.29	1.71	1.52	1.99	76.78	69.39	17.46	20.91	90.394	8.9			
252	do.	353	do.	1905 (1)	9.36	90.64	3.04	13.94	9.76	2.29	1.71	1.52	1.99	76.78	69.39	17.46	20.91	90.394	8.9			
253	do.	353	do.	1905 (1)	9.45	90.51	2.65	16.50	11.55	2.29	1.71	1.52	1.99	76.78	69.39	17.46	20.91	90.394	8.9			
254	do.	187	Sweden.	Original	9.36	90.64	3.04	12.37	8.66	2.29	1.71	1.52	1.99	76.78	69.39	17.46	20.91	90.394	8.9			
255	do.	187	Kans.	1905 (4)	9.45	90.51	2.65	16.50	11.55	2.29	1.71	1.52	1.99	76.78	69.39	17.46	20.91	90.394	8.9			
256	do.	187	S. Dak.	1904 (2)	9.70	91.30	2.80	14.81	10.37	2.59	1.71	1.52	1.99	76.78	69.39	17.46	20.91	90.394	8.9			
257	do.	187	do.	1905 (3)	8.54	90.46	2.37	15.75	11.02	2.44	1.90	1.90	1.90	76.78	69.39	17.46	20.91	90.394	8.9			
258	do.	187	Wash.	1903 (1)	9.26	90.74	3.11	16.12	11.26	2.59	1.71	1.52	1.99	76.78	69.39	17.46	20.91	90.394	8.9			
259	do.	187	Cal.	1902 (2)	9.52	90.49	2.76	9.26	6.48	1.52	1.78	1.60	1.60	76.78	69.39	17.46	20.91	90.394	8.9			

1182	Klitzing	Bavaria	Original	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100	101	102	103	104	105	106	107	108	109	110	111	112	113	114	115	116	117	118	119	120	121	122	123	124	125	126	127	128	129	130	131	132	133	134	135	136	137	138	139	140	141	142	143	144	145	146	147	148	149	150	151	152	153	154	155	156	157	158	159	160	161	162	163	164	165	166	167	168	169	170	171	172	173	174	175	176	177	178	179	180	181	182	183	184	185	186	187	188	189	190	191	192	193	194	195	196	197	198	199	200	201	202	203	204	205	206	207	208	209	210	211	212	213	214	215	216	217	218	219	220	221	222	223	224	225	226	227	228	229	230	231	232	233	234	235	236	237	238	239	240	241	242	243	244	245	246	247	248	249	250	251	252	253	254	255	256	257	258	259	260	261	262	263	264	265	266	267	268	269	270	271	272	273	274	275	276	277	278	279	280	281	282	283	284	285	286	287	288	289	290	291	292	293	294	295	296	297	298	299	300	301	302	303	304	305	306	307	308	309	310	311	312	313	314	315	316	317	318	319	320	321	322	323	324	325	326	327	328	329	330	331	332	333	334	335	336	337	338	339	340	341	342	343	344	345	346	347	348	349	350	351	352	353	354	355	356	357	358	359	360	361	362	363	364	365	366	367	368	369	370	371	372	373	374	375	376	377	378	379	380	381	382	383	384	385	386	387	388	389	390	391	392	393	394	395	396	397	398	399	400	401	402	403	404	405	406	407	408	409	410	411	412	413	414	415	416	417	418	419	420	421	422	423	424	425	426	427	428	429	430	431	432	433	434	435	436	437	438	439	440	441	442	443	444	445	446	447	448	449	450	451	452	453	454	455	456	457	458	459	460	461	462	463	464	465	466	467	468	469	470	471	472	473	474	475	476	477	478	479	480	481	482	483	484	485	486	487	488	489	490	491	492	493	494	495	496	497	498	499	500	501	502	503	504	505	506	507	508	509	510	511	512	513	514	515	516	517	518	519	520	521	522	523	524	525	526	527	528	529	530	531	532	533	534	535	536	537	538	539	540	541	542	543	544	545	546	547	548	549	550	551	552	553	554	555	556	557	558	559	560	561	562	563	564	565	566	567	568	569	570	571	572	573	574	575	576	577	578	579	580	581	582	583	584	585	586	587	588	589	590	591	592	593	594	595	596	597	598	599	600	601	602	603	604	605	606	607	608	609	610	611	612	613	614	615	616	617	618	619	620	621	622	623	624	625	626	627	628	629	630	631	632	633	634	635	636	637	638	639	640	641	642	643	644	645	646	647	648	649	650	651	652	653	654	655	656	657	658	659	660	661	662	663	664	665	666	667	668	669	670	671	672	673	674	675	676	677	678	679	680	681	682	683	684	685	686	687	688	689	690	691	692	693	694	695	696	697	698	699	700	701	702	703	704	705	706	707	708	709	710	711	712	713	714	715	716	717	718	719	720	721	722	723	724	725	726	727	728	729	730	731	732	733	734	735	736	737	738	739	740	741	742	743	744	745	746	747	748	749	750	751	752	753	754	755	756	757	758	759	760	761	762	763	764	765	766	767	768	769	770	771	772	773	774	775	776	777	778	779	780	781	782	783	784	785	786	787	788	789	790	791	792	793	794	795	796	797	798	799	800	801	802	803	804	805	806	807	808	809	810	811	812	813	814	815	816	817	818	819	820	821	822	823	824	825	826	827	828	829	830	831	832	833	834	835	836	837	838	839	840	841	842	843	844	845	846	847	848	849	850	851	852	853	854	855	856	857	858	859	860	861	862	863	864	865	866	867	868	869	870	871	872	873	874	875	876	877	878	879	880	881	882	883	884	885	886	887	888	889	890	891	892	893	894	895	896	897	898	899	900	901	902	903	904	905	906	907	908	909	910	911	912	913	914	915	916	917	918	919	920	921	922	923	924	925	926	927	928	929	930	931	932	933	934	935	936	937	938	939	940	941	942	943	944	945	946	947	948	949	950	951	952	953	954	955	956	957	958	959	960	961	962	963	964	965	966	967	968	969	970	971	972	973	974	975	976	977	978	979	980	981	982	983	984	985	986	987	988	989	990	991	992	993	994	995	996	997	998	999	1000	1001	1002	1003	1004	1005	1006	1007	1008	1009	1010	1011	1012	1013	1014	1015	1016	1017	1018	1019	1020	1021	1022	1023	1024	1025	1026	1027	1028	1029	1030	1031	1032	1033	1034	1035	1036	1037	1038	1039	1040	1041	1042	1043	1044	1045	1046	1047	1048	1049	1050	1051	1052	1053	1054	1055	1056	1057	1058	1059	1060	1061	1062	1063	1064	1065	1066	1067	1068	1069	1070	1071	1072	1073	1074	1075	1076	1077	1078	1079	1080	1081	1082	1083	1084	1085	1086	1087	1088	1089	1090	1091	1092	1093	1094	1095	1096	1097	1098	1099	1100	1101	1102	1103	1104	1105	1106	1107	1108	1109	1110	1111	1112	1113	1114	1115	1116	1117	1118	1119	1120	1121	1122	1123	1124	1125	1126	1127	1128	1129	1130	1131	1132	1133	1134	1135	1136	1137	1138	1139	1140	1141	1142	1143	1144	1145	1146	1147	1148	1149	1150	1151	1152	1153	1154	1155	1156	1157	1158	1159	1160	1161	1162	1163	1164	1165	1166	1167	1168	1169	1170	1171	1172	1173	1174	1175	1176	1177	1178	1179	1180	1181	1182	1183	1184	1185	1186	1187	1188	1189	1190	1191	1192	1193	1194	1195	1196	1197	1198	1199	1200	1201	1202	1203	1204	1205	1206	1207	1208	1209	1210	1211	1212	1213	1214	1215	1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TABLE XLIII.—Barley—individual analyses—Continued.

Laboratory No.	Variety.	Grain investigation No.	Locality.	Year and crop.	Water.		Dry matter.	Ash.	Protein (N X 6.25).			Fat.		Crude fiber.		Carbohydrate.		Total production value.		Nutritive ratio.	
									Total.	Digestible.	Production value in flesh.	Total.	Digestible.	Production value in flesh.	Total.	Digestible.	Production value in flesh.	Calories.			
1294	Fan.	255	S. Dak.	1905 (2)	9.59	90.41	3.03	14.62	10.23	2.40	1.59	0.84	1.59	1.66	0.41	75.66	67.70	17.23	20.88	90.264	8.3
1295	Marriott.	261	do.	1905 (2)	9.27	90.73	3.15	14.25	9.97	2.34	1.52	0.77	1.52	2.45	0.61	73.66	67.76	17.30	20.86	88.449	7.3
1296	do.	261	Tex.	1905 (1)	9.01	91.00	3.36	11.69	1.92	1.92	1.46	0.77	1.46	2.36	0.36	67.76	70.17	17.37	20.64	90.178	9.3
1297	Manchuria.	170	S. Dak.	1905 (3)	9.58	90.42	2.99	13.25	9.27	2.18	1.65	0.77	1.65	2.14	0.53	76.24	70.17	17.38	20.86	90.227	8.1
1298	do.	170	do.	1904 (2)	8.87	91.13	3.27	13.62	9.53	2.24	1.51	0.80	1.51	2.34	0.36	76.24	68.46	16.98	20.79	88.621	7.7
1299	do.	170	Alaska.	1903	8.81	91.69	3.34	9.75	6.82	1.60	1.71	1.52	1.71	2.33	0.58	74.44	71.80	17.81	20.79	89.875	11.4
1300	Sesolk.	80	Kans.	1905 (4)	8.93	91.07	3.40	15.06	10.54	2.48	1.41	0.85	1.41	1.90	0.47	73.66	67.97	16.96	20.66	90.313	7.0
1301	Giant Winter.	30	Oreg.	1904 (2)	8.92	91.06	3.24	9.37	6.56	1.54	1.86	1.06	1.86	2.23	0.74	74.72	72.94	19.33	21.72	93.815	12.4
1302	Hull-less.	197	Wash.	1903 (1)	8.94	91.06	3.22	18.09	13.08	3.07	2.31	1.06	2.31	2.76	0.91	72.80	66.27	16.84	21.22	91.734	5.6
1303	Hull-less Hankin.	321	Kans.	1903	8.94	91.36	3.58	20.03	14.44	3.39	1.94	1.73	1.94	2.73	0.93	72.80	67.97	16.84	21.22	91.734	5.6
1304	Hull-less Nepal.	352	S. Dak.	1905 (2)	9.45	90.55	2.23	15.56	10.80	2.56	1.81	0.85	1.81	2.73	0.90	72.80	67.97	16.84	21.22	92.206	7.0
1305	Hull-less Beardless.	321	do.	1905 (2)	9.41	90.59	2.34	16.19	11.33	2.66	1.99	0.87	1.99	2.67	0.88	72.80	67.97	16.84	21.22	92.206	6.7
1306	Mexican Beardless.	202	Arabia.	Original.	9.30	90.70	2.80	13.06	9.14	2.15	1.87	1.66	1.87	2.67	0.88	72.80	67.97	16.84	21.21	91.001	9.0
1307	Black Arabian.	263	England.	do.	9.44	90.56	2.74	13.75	9.62	2.28	1.99	1.10	1.99	2.67	0.88	72.80	67.97	16.84	21.21	91.001	9.0
1308	do.	264	do.	do.	9.51	90.49	2.74	13.75	9.62	2.26	2.35	1.10	2.35	2.67	0.88	72.80	67.97	16.84	21.21	91.001	9.0
1309	do.	265	do.	do.	9.46	90.54	2.99	13.75	9.62	2.16	2.11	1.10	2.11	2.67	0.88	72.80	67.97	16.84	21.21	91.001	9.0
1310	do.	266	do.	do.	9.44	90.56	2.63	10.37	9.18	1.98	1.59	1.42	1.59	2.67	0.88	72.80	67.97	16.84	21.21	91.001	9.0
1311	do.	267	Ariz.	1905 (1)	10.11	89.80	3.24	15.31	10.72	2.32	1.85	1.65	1.85	2.67	0.88	72.80	67.97	16.84	21.21	92.771	11.0
1312	do.	268	Kans.	1905 (2)	9.38	90.62	3.24	17.62	12.33	2.90	2.07	1.65	2.07	2.67	0.88	72.80	67.97	16.84	21.21	92.771	11.0
1313	do.	269	do.	1905 (2)	9.96	90.04	3.24	17.62	12.33	2.90	2.32	1.85	2.32	2.67	0.88	72.80	67.97	16.84	21.21	92.771	11.0
1314	do.	264	Wash.	1905 (2)	9.06	90.94	2.98	9.13	6.39	1.50	2.22	1.08	2.22	2.67	0.88	72.80	67.97	16.84	21.21	92.771	11.0
1315	do.	265	do.	1905 (2)	9.33	90.67	3.21	12.75	8.92	2.16	2.16	1.08	2.16	2.67	0.88	72.80	67.97	16.84	21.21	92.771	11.0
1316	do.	265	Kans.	1905 (2)	9.73	90.27	3.39	17.37	12.16	2.86	2.16	1.08	2.16	2.67	0.88	72.80	67.97	16.84	21.21	92.771	11.0
1317	Hanna.	203	N. Dak.	1902 (1)	9.65	90.35	3.17	16.69	11.68	2.74	1.95	1.74	1.95	2.67	0.88	72.80	67.97	16.84	21.21	91.388	7.9
1318	do.	203	do.	1902 (2)	9.11	90.89	2.67	13.81	9.67	2.13	2.15	1.01	2.15	2.67	0.88	72.80	67.97	16.84	21.21	91.388	7.9
1319	do.	203	do.	1902 (3)	9.11	90.81	3.41	11.62	8.13	1.97	2.25	1.01	2.25	2.67	0.88	72.80	67.97	16.84	21.21	91.388	7.9
1320	do.	203	S. Dak.	1903 (3)	9.58	90.42	3.41	14.87	10.41	2.45	2.37	1.11	2.37	2.67	0.88	72.80	67.97	16.84	21.21	91.388	7.9
1321	do.	203	do.	1903 (2)	9.24	90.76	3.23	15.62	10.93	2.57	1.73	1.11	1.73	2.67	0.88	72.80	67.97	16.84	21.21	91.388	7.9
1322	do.	203	do.	1904 (2)	9.11	90.80	3.13	19.94	13.61	3.22	2.19	1.08	2.19	2.67	0.88	72.80	67.97	16.84	21.21	91.388	7.9
1323	do.	203	do.	1904 (3)	9.11	90.14	3.31	13.31	9.31	2.29	1.97	1.73	1.97	2.67	0.88	72.80	67.97	16.84	21.21	91.388	7.9
1324	do.	203	do.	1905 (3)	9.11	90.80	3.30	13.31	9.31	2.29	1.97	1.73	1.97	2.67	0.88	72.80	67.97	16.84	21.21	91.388	7.9
1325	do.	203	do.	1905 (4)	9.82	90.18	3.27	12.37	6.66	2.04	1.68	1.75	1.68	2.67	0.88	72.80	67.97	16.84	21.21	91.388	7.9
1326	do.	203	Idaho.	1905 (4)	9.46	90.52	3.27	11.01	7.70	1.81	1.68	1.75	1.68	2.67	0.88	72.80	67.97	16.84	21.21	91.388	7.9
1327	do.	203	Minn.	1901 (1)	8.75	90.46	3.04	15.81	11.07	2.63	1.81	1.68	1.81	2.67	0.88	72.80	67.97	16.84	21.21	91.388	7.9
1328	do.	203	do.	1902 (1)	8.75	91.25	3.04	17.19	12.03	2.83	1.81	1.68	1.81	2.67	0.88	72.80	67.97	16.84	21.21	91.388	7.9
1329	do.	203	Cal.	1902 (1)	9.14	90.80	2.97	14.06	9.84	2.60	1.81	1.68	1.81	2.67	0.88	72.80	67.97	16.84	21.21	91.388	7.9
1330	do.	203	do.	1902 (1)	9.20	90.86	2.62	15.81	11.07	2.60	1.81	1.68	1.81	2.67	0.88	72.80	67.97	16.84	21.21	91.388	7.9

1110	do.	1003 (1)	9.75	90.03	2.87	10.19	7.13	1.69	2.05	1.89	.95	4.79	1.57	39	80.16	73.74	18.29	21.31	92.123	11.7
1111	do.	1004 (3)	9.35	90.65	2.89	9.73	6.82	1.69	2.05	1.89	.96	5.23	1.77	38	80.17	73.75	18.29	21.31	91.963	11.7
1112	do.	1002 (1)	9.39	90.61	2.89	9.04	6.98	1.64	1.89	1.59	.79	4.44	1.73	38	81.01	74.52	18.48	21.32	91.963	11.7
1113	do.	1003 (3)	9.77	90.23	2.90	13.37	6.98	2.72	1.89	1.59	.85	4.50	1.49	35	80.40	74.23	18.48	21.32	91.972	8.2
1114	do.	1003 (3)	9.31	90.69	2.42	10.44	7.31	1.72	1.81	1.61	.85	4.64	1.53	38	80.90	74.23	18.41	21.36	92.339	10.9
1115	do.	1001 (1)	9.38	90.62	3.07	13.00	8.71	2.14	2.21	1.97	.95	4.52	1.49	37	77.20	71.52	17.91	21.19	91.479	8.5
1116	do.	1002 (1)	9.32	90.68	3.03	12.44	8.71	2.03	2.02	1.80	.96	4.86	1.64	41	77.53	71.54	17.99	21.19	91.218	8.8
1117	do.	1005 (3)	10.01	89.99	3.58	17.25	12.07	2.64	1.91	1.70	.86	4.66	1.54	38	72.60	66.79	16.56	20.67	89.356	8.0
1118	do.	1004 (1)	9.28	90.72	2.91	12.87	9.01	2.12	1.83	1.63	.86	4.62	1.52	38	77.77	71.58	17.74	21.10	91.215	8.5
1119	do.	1003 (1)	9.16	90.84	3.13	14.46	10.12	2.36	1.96	1.74	.91	5.13	1.69	42	75.22	69.20	17.16	20.87	90.221	7.4
1120	do.	1004 (2)	9.21	90.79	2.67	13.00	9.10	2.14	1.96	1.74	.91	5.03	1.68	41	77.37	71.18	17.65	21.11	91.258	8.4
1121	do.	1003 (2)	9.84	90.16	3.26	16.31	11.42	2.69	2.00	1.78	.94	4.81	1.69	39	73.62	67.73	16.80	20.81	89.963	6.4
1122	do.	1005 (2)	9.24	90.76	3.02	13.62	9.53	2.24	1.92	1.71	.90	4.78	1.58	39	78.66	70.52	17.49	21.02	90.869	8.0
1123	do.	1003 (1)	9.73	90.27	3.01	12.00	8.40	1.97	2.08	1.89	.97	4.87	1.61	40	78.04	71.79	17.80	21.14	91.388	9.2

TABLE XLIV.—*Proso—individual analyses.*

[Pounds per hundred pounds of dry matter.]

Laboratory No.	Variety.	Grain Investigation No.	Locality.	Year and crop.	Protein (N X 6.25).			Fat.		Crude fiber.		Carbohydrate.		Total production value.	
					Total.	Digestible.	Production value—grain in flesh.	Digestible.	Production value—grain in flesh.	Total.	Digestible.	Total.	Digestible.	Calories.	Nutritive ratio.
1037	Red Orenburg.	21	Kans.	1905 (2)	12.69	6.24	1.47	3.51	2.70	1.42	1.50	71.05	60.53	83,806	11.7
1002	do.	21	do.	1904 (1)	13.12	6.45	1.51	3.32	2.55	1.34	1.32	72.09	61.42	83,846	11.7
1003	do.	21	do.	1904 (2)	17.94	8.83	2.07	3.18	2.45	1.29	1.58	65.93	56.17	81,575	7.7
1004	do.	21	Iowa.	1903 (1)	13.87	6.82	1.60	3.00	2.31	1.22	1.35	71.57	60.98	83,520	10.5
1005	do.	21	do.	1904 (2)	13.44	6.61	1.55	3.33	2.31	1.35	1.57	71.16	60.63	84,298	11.0
1006	do.	21	do.	1904 (2)	12.62	6.21	1.46	3.77	2.90	1.53	1.36	71.98	61.32	84,558	11.9
1007	do.	21	Nebr.	1905 (2)	11.06	5.44	1.28	3.52	2.71	1.43	1.35	73.78	62.86	84,947	13.7
1008	do.	21	do.	1905 (2)	12.50	6.15	1.45	3.26	2.51	1.32	1.64	68.58	59.22	82,099	11.6
1009	do.	21	N. Dak.	1904 (1)	12.37	6.09	1.43	3.54	2.72	1.43	1.84	69.51	59.22	83,823	11.9
1010	do.	21	do.	1904 (2)	10.94	5.38	1.26	3.36	2.58	1.36	1.48	73.66	62.76	84,990	13.9
1011	do.	21	Idaho.	1905 (2)	11.44	5.63	1.32	2.98	2.29	1.20	1.59	72.28	61.58	83,780	13.1
1012	do.	21	do.	1904 (2)	11.81	5.81	1.37	2.93	2.25	1.18	1.55	71.76	61.14	83,663	12.5
1013	do.	21	do.	1904 (2)	14.81	7.29	1.71	3.43	2.68	1.39	1.55	73.76	62.84	84,126	9.9
1014	do.	21	do.	1905 (3)	15.81	8.11	1.21	3.99	2.53	1.21	1.43	71.86	63.74	85,552	14.6
1015	do.	21	do.	1904 (2)	10.44	5.14	1.21	3.33	2.56	1.35	1.54	72.45	61.74	85,045	9.0
1016	do.	21	Okl.	1905 (2)	12.09	5.90	1.36	3.29	2.53	1.33	1.67	71.58	60.98	84,601	12.5
1017	do.	21	do.	1904 (1)	11.87	5.90	1.37	3.48	2.68	1.41	1.58	73.76	62.84	84,947	11.2
1018	do.	26	Nebr.	1904 (1)	13.00	6.41	1.57	3.18	2.45	1.28	1.52	73.76	62.84	84,428	13.6
1019	do.	26	Kans.	1905 (2)	11.00	5.41	1.20	3.17	2.44	1.22	1.52	73.76	62.84	81,532	9.2
1020	do.	26	do.	1904 (1)	15.25	7.50	1.76	3.02	2.32	1.22	1.78	72.13	61.45	83,520	14.9
1021	do.	26	Idaho.	1905 (2)	10.00	4.92	1.16	2.32	2.32	1.20	1.70	72.13	61.45	80,840	8.2
1022	do.	26	do.	1904 (1)	16.87	8.30	1.93	2.28	2.28	1.20	1.72	70.13	59.75	82,872	12.4
1023	do.	26	Wash.	1904 (1)	16.87	8.30	1.93	2.28	2.28	1.20	1.72	70.13	59.75	82,872	12.4
1024	do.	26	do.	1905 (2)	15.94	8.11	1.84	2.97	2.40	1.26	1.72	70.13	59.75	82,872	12.4
1025	do.	26	do.	1905 (2)	15.94	8.11	1.84	2.97	2.40	1.26	1.72	70.13	59.75	82,872	12.4
1026	do.	26	Tex.	1904 (1)	11.94	5.87	1.34	2.88	2.28	1.16	1.85	70.13	59.75	82,872	12.4
1027	Black Voronezh.	27	Nebr.	1904 (1)	11.37	5.59	1.32	3.70	2.85	1.50	1.35	73.38	62.92	83,520	12.3
1028	do.	27	Idaho.	1904 (1)	11.34	5.63	1.33	3.41	2.62	1.38	1.35	73.38	62.92	83,520	12.3
1029	do.	27	do.	1904 (1)	11.69	5.75	1.35	3.45	2.60	1.37	1.35	73.38	62.92	83,520	12.3
1030	do.	27	do.	1904 (1)	12.87	6.33	1.39	3.38	2.65	1.37	1.35	73.38	62.92	83,520	12.3
1031	do.	27	Okl.	1904 (1)	12.87	6.33	1.39	3.38	2.65	1.37	1.35	73.38	62.92	83,520	12.3
1032	do.	27	Wash.	1903 (1)	12.00	5.90	1.39	3.19	2.45	1.20	1.46	71.86	61.24	83,946	12.4
1033	do.	27	Tex.	1903 (1)	16.37	8.05	1.80	3.14	2.41	1.27	1.46	71.86	61.24	83,946	12.4
1034	do.	27	do.	1905 (2)	12.75	6.27	1.47	3.22	2.49	1.30	1.49	71.86	61.24	83,946	12.4

TABLE XLV.—*Sorghum—individual analyses.*

[Pounds per hundred pounds of dry matter.]

TABLE XLV.— <i>Sorghum—individual analyses.</i>																				
[Pounds per hundred pounds of dry matter.]																				
Laboratory No.	Variety.	Grain investigation No.	Water.	Dry matter.	Ash.	Protein (N X 6.25).				Fat.		Crude fiber.		Carbohydrate.			Total production value.		Nutritive ratio.	
						Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Total.	Digestible.	Production value—gain in flesh.	Gain in flesh.	Calories.				
798	Edra.....	8815	10.91	89.09	2.18	11.44	7.44	1.75	4.34	3.04	1.60	1.73	0.88	22	80.31	73.08	18.12	21.69	83,766	10.9
799	Gidlep-jowar.....	9655	11.02	88.98	1.64	10.13	6.58	1.55	3.30	2.31	1.22	1.57	.80	20	83.36	75.86	18.61	21.78	84,155	12.5
770	Dayel-jowar.....	9656	11.35	88.65	1.75	8.87	5.77	1.36	2.41	1.69	1.89	1.66	.85	20	85.31	77.64	19.25	21.71	83,852	14.3
771	Natal.....	10327	10.91	89.09	1.60	14.50	9.42	2.21	2.97	2.08	1.09	1.94	.99	25	79.99	71.83	17.83	21.38	82,426	8.2
772	Duggara.....	10612	11.35	88.65	2.04	13.81	8.98	2.11	3.52	2.46	1.29	1.76	.85	27	79.96	72.76	18.05	21.66	83,636	9.6
777	White Milo.....	2523	12.80	87.20	1.67	11.12	7.23	1.70	2.65	1.85	1.07	2.12	1.08	21	82.44	75.02	18.60	21.54	83,253	11.1
779	White Kafr.....	1963	12.10	87.90	1.97	12.94	8.41	1.98	2.82	1.97	1.03	2.32	1.18	29	78.65	72.75	18.04	21.54	82,253	9.3
780	do.....	13317	12.55	87.45	1.65	11.56	7.51	1.76	3.33	2.33	1.23	1.78	.91	23	81.08	74.32	18.43	21.87	83,583	10.7
781	Red Kafr.....	13318	12.31	87.69	1.51	10.06	6.54	1.54	3.73	2.61	1.37	1.66	.79	20	83.13	75.65	18.76	21.65	84,544	12.5
782	Dwarf Milo.....	13565	11.84	88.16	1.54	12.69	8.25	1.94	3.41	2.39	1.26	1.65	.84	21	80.71	73.45	18.22	21.63	83,506	9.7

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Issued January 25, 1909.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY—BULLETIN No. 121.
H. W. WILEY, Chief of Bureau.

FOOD LEGISLATION DURING THE YEAR
ENDED JUNE 30, 1908.

BY

W. D. BIGELOW,
Chief, Division of Foods,

WITH THE COLLABORATION OF

N. A. PARKINSON.



WASHINGTON:
GOVERNMENT PRINTING OFFICE.

1909.

LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., November 30, 1908.

SIR: I have the honor to transmit for your inspection and approval a compilation of the food legislation during the year ended June 30, 1908. I recommend its publication as Bulletin 121 of the Bureau of Chemistry, supplementing Bulletin 112, Parts I and II, Food Legislation during the Year ended June 30, 1907; Bulletin 104, covering the year ended June 30, 1906, and Bulletin 69, Revised, Parts I to IX, covering all legislation prior to July 1, 1905. This compilation is of special value at this time, when so many changes are being made in the State laws tending toward uniformity with the National food law, and in view of the close cooperation between State and Federal food officials.

Respectfully,

H. W. WILEY,
Chief of Bureau.

Hon. JAMES WILSON,
Secretary of Agriculture.

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FOOD LEGISLATION DURING THE YEAR ENDED JUNE 30, 1908.

FEDERAL LAWS.

During the fiscal year ended June 30, 1908, no Federal food laws were passed with the exception of the amendment to the tea law, which is printed herewith. The following orders, regulations, and decisions issued by the Bureau of Animal Industry and the Board of Food and Drug Inspection of the United States Department of Agriculture and by the Bureau of Internal Revenue of the United States Treasury Department are enumerated as a matter of record, but are not reprinted:

Bureau of Animal Industry: B. A. I. Order No. 147, Regulations Prescribed in Regard to Renovated Butter in Accordance with the Act of Congress Approved May 9, 1902 (July 11, 1907). B. A. I. Order No. 150, Regulations Governing the Meat Inspection of the United States Department of Agriculture (effective April 1, 1908). Amendment to B. A. I. Order No. 150, Amendment to Section 19 of Regulation 25, Exempting Shipments of Certain Inedible Grease, Tallow, or Other Fat from the Provision Requiring the Denaturing of Inedible Products (April 24, 1908). B. A. I. Meat Inspection Rulings—2 A, Colors (September 2, 1907). B. A. I. Meat Inspection Rulings—3 A, Notice Regarding the Enforcement of that Portion of Paragraph 5 of Section 19 of Regulation 25 of B. A. I. Order 150, Relating to the Denaturing of Inedible Grease, Tallow, and Other Fat (April 6, 1908).

Board of Food and Drug Inspection: Food Inspection Decision 74, Certificates for Imported Meats and Meat-Food Products of Cattle, Sheep, Swine, and Goats. 75, The Labeling of Mixtures of Cane and Maple Sirups. 76, Dyes, Chemicals, and Preservatives in Foods. 77, Certificate and Control of Dyes Permissible for Use in Coloring Foods and Foodstuffs. 78, The Use of Labels after October 1, 1907. 79, Collection of Samples. 80, Glazed Coffee. 81, Labeling of Caramels. 82, Labeling of Coffee Produced in the Dutch East Indies. 83, The Issue of a Guaranty Based upon a Former Guaranty. 84, Amendments to Regulations 17 and 19. 85, Labeling of Bitters. 86, Original Packages: Interpretation of Regulation 2 of Rules and Regulations for the Enforcement of the Food and

Drugs Act. 87, Labeling of Corn Sirup. 88, Private Importations. 89, Amendment to Food Inspection Decision 76, Relating to the Use in Foods of Benzoate of Soda and Sulphur Dioxid. 90, The Labeling of Foods and Medicinal Mixtures for Stock and Poultry. 91, The Labeling of Mocha Coffee. 92, The Use of Copper Salts in the Greening of Foods. 93, Amendment to Regulation 34. 94, The Labeling of Medicinal and Table Waters. 95, The Use of Neutral Spirits Distilled from Beet Sugar Molasses in the Preparation of Whisky Compounds and Imitation Whiskies. 96, Serial Number Guaranty.

Bureau of Internal Revenue: Regulation No. 9, Revised July, 1907, Revised Regulations Concerning Oleomargarine, also Adulterated Butter and Process or Renovated Butter.

TEA.

SEC. 1. *Importation of inferior tea prohibited; proviso.* From and after May first, eighteen hundred and ninety-seven, it shall be unlawful for any person or persons or corporation to import or bring into the United States any merchandise as tea which is inferior in purity, quality, and fitness, for consumption to the standards provided in section three ^a of this act, and the importation of all such merchandise is hereby prohibited. *Provided*, That nothing herein shall affect or prevent the importation into the United States, under such regulations as the Secretary of the Treasury may prescribe, of any merchandise as tea which may be inferior in purity, quality, and fitness for consumption to the standards established by the Secretary of the Treasury, or of any tea waste, tea siftings, or tea sweepings, for the sole purpose of manufacturing theine, caffeine, or other chemical products whereby the identity and character of the original material is entirely destroyed or changed; and that importers and manufacturers who import or bring into the United States such tea, tea waste, tea siftings, or tea sweepings shall give suitable bond, to be approved as to amount and securities by the Secretary of the Treasury, conditioned that said imported material shall be only used for the purposes herein provided, under such regulations as may be prescribed by the Secretary of the Treasury.—*As amended May 16, 1908. Statutes of the United States of America, 1907-1908, Part 1, ch. 170, p. 163.*

Approved March 2, 1897. United States Statutes at Large, 1895-1897, vol. 29, ch. 358, pp. 604-607.

^a See Bul. 69, Revised, Part I, p. 7.

CANADA.

The statutes of Canada were not available and only those laws are included of which copies could be procured from the enforcing officer.

MEAT AND CANNED GOODS.

SEC. 1. *Short title.* This Act may be cited as *The Meat and Canned Foods Act*.

SEC. 2. *Definitions.* In this Act, unless the context otherwise requires,

(a) "carcases" means the carcasses of cattle, swine, sheep, goats or poultry;

(b) "establishment" means any abattoir, packing house, or other premises in which such animals are slaughtered, or in which any parts thereof or products thereof, or fish, or fruit, or vegetables, are prepared for food for export or are stored for export;

(c) "export" means export out of Canada, or out of the province in which the establishment is situated to another province;

(d) "food" includes every article used for food or drink by man, and every ingredient intended for mixing with the food or drink of man for any purpose;

(e) "inspector" means an inspector appointed under this Act;

(f) "Minister" means the Minister of Agriculture;

(g) "regulations" means regulations made under the provisions of this Act.

SEC. 3. *Inspection of animals.* All animals intended for slaughter in any establishment shall be inspected as provided by the regulations.

2. No animal shall be allowed to enter the parts of an establishment where slaughtering is carried on, unless it has undergone such inspection and been found to be healthy and fit for food.

3. Every animal affected, or suspected of being affected, with contagious or other disease, shall be slaughtered under the supervision of the inspector and be disposed of as provided by the regulations.

SEC. 4. *Inspection of carcasses.* All carcasses and portions thereof of all animals, wherever slaughtered, intended for export, shall be inspected as provided by the regulations.

SEC. 5. *Slaughtering by farmers and retail butchers.* Unless the Minister otherwise directs, upon the report of an inspector, animals owned by farmers and slaughtered by them on their own premises, and animals slaughtered by retail butchers on their own premises, shall not be subject to inspection under the provisions of this Act.

SEC. 6. *Healthy carcasses; marks on.* Every carcass, or portion thereof, found to be healthy and fit for food, shall be marked by the inspector in such manner as is provided by the regulations; and the carcass, or portion thereof, may then be dealt with as the owner thereof sees fit, subject to the further supervision of the inspector.

SEC. 7. *Inspection and marking of meat products.* Every carcass or portion or product thereof prepared for food in any establishment and packed in cans or similar receptacles, or in any package whatever, shall be subject to inspection during the whole course of preparation and packing; and after all the requirements of this Act regarding inspection have been complied with, and not until then, all such packages shall be marked by the inspector in such manner as is provided by the regulations.

SEC. 8. *Re-inspection.* The inspector may at any time re-inspect a carcass, or any portion or product thereof, in order to ascertain whether, subsequently to the first inspection thereof, it has undergone decomposition, or has otherwise deteriorated, or has been tampered with or adulterated by the use of preservatives or otherwise.

2. Every carcass, or portion or product thereof, sent out of an establishment, and returned thereto for any purpose, shall not be again sent out therefrom without re-inspection.

SEC. 9. *Unhealthy meat, disposal of.* Every carcass, or portion or product thereof, found, upon inspection or re-inspection, to be unhealthy or unfit for food, or which contains such ingredients or preservatives as may render it unfit for food, shall be marked by the inspector in such manner as is provided by the regulations, and shall thereupon be deemed to be condemned as unfit for food and shall be disposed of as provided by the regulations.

SEC. 10. *Sale, etc., of unhealthy meat.* Any person slaughtering, or permitting the slaughtering of, animals and selling, or offering for sale or transportation, for export a carcass, or any portion or product thereof, which is unhealthy or unfit for food is guilty of an indictable offence and liable to one year's imprisonment.

2. Every one who is convicted of this offence after a previous conviction for the same crime shall be liable to two years' imprisonment.

SEC. 11. *Exemption from inspection.* The Governor in Council may, upon application of the owner thereof, exempt any establishment from the operation of the provisions of sections 3 and 4, and of sections 6 to 10, both inclusive, of this Act.—*As amended June 16, 1908. 7-8 Edward VII, ch. 47.*

SEC. 12. *Inspection and marking of packages.* All articles prepared for food in any establishment and packed in cans or similar receptacles, or in any package whatever, shall be subject to inspection during the whole course of preparation and packing; and all such packages shall be marked with—

(a) the initials of the Christian names, the full surname, and the address, or, in the case of a firm or corporation, the firm or corporate name and address, of the packer; or of the first dealer obtaining them direct from the packer who sells or offers the said articles for sale; and such dealer shall, upon the request of an inspector appointed under this Act, disclose the name of the packer of such article.—*As amended June 16, 1908. 7-8 Edward VII, ch. 47.*

(b) a true and correct description of the contents of the package:

Provided, however, that if it be established to the satisfaction of the Governor in Council that such marking would hinder the sale of any of said articles in the British or foreign markets, he may exempt such articles from the provisions of this section.

SEC. 13. *Fish, fruit and vegetables.* All fish, fruit, or vegetables used in any establishment where these articles are prepared for export, shall be sound, wholesome, and fit for food; and any such articles or products thereof found in the said establishment unsound or unwholesome shall be confiscated and destroyed as provided by the regulations.

SEC. 14. *Sanitary conditions.* An inspection and close supervision of the sanitary conditions of any establishments shall be maintained as provided by the regulations.

2. The inspector shall refuse to inspect or mark articles in any establishment where the sanitary conditions are not in accordance with the regulations.

SEC. 15. *Withdrawal of inspector and closing of establishment for violation of Act, etc.* In the event of the provisions of this Act, or any regulations, or the lawful instruction of an inspector not being complied with in any establishment, the Minister may withdraw the inspector therefrom, and may refuse to it the inspection, marking, and certification of the articles prepared therein, and may cause the establishment to be closed.

SEC. 15A. *Sale in violation of Act.* No person shall offer or expose or have in his possession for sale any article subject to inspection under this Act unless all the requirements thereof respecting the said article have been complied with.—*Added June 16, 1908. 8-7 Edward VII, ch. 47.*

SEC. 16. *Export of uninspected articles.* No person shall offer or accept for export, or shall export, any articles subject to inspection under this Act, unless its requirements regarding inspection and marking have been complied with in respect to such articles.

2. No clearance shall be granted to any vessel carrying any carcases, or any portions or products thereof, unless they are duly marked in accordance with the provisions of this Act.

3. The provisions of this section shall not apply to meats intended for consumption on board the vessels by which they are shipped from a Canadian port.

4. At the request of the owner of any establishment, the inspector in charge thereof shall issue certificates of inspection for any carcases or portions or products thereof intended for export. Such certificates shall be in such form as is provided by the regulations.

5. Notwithstanding anything in this section, the Governor in Council may, whenever it is deemed necessary or advisable to do so, authorize the export of any such articles without inspection.

SEC. 17. *False marking as to name, weight, and date.* No article subject to inspection under this Act shall be offered or sold for export, or exported, under any name intended or calculated to deceive as to its true nature.

2. No package containing any article subject to inspection under this Act shall be marked with any label, brand or mark which falsely represents the quantity or weight or contents of such package.

3. No package containing any article subject to inspection under this Act shall be marked with any label, brand or mark which falsely represents the date when the articles or goods contained therein were packed.—*As amended June 16, 1908. 7-8 Edward VII, ch. 47.*

SEC. 18. *Tampering with marks.* Every person who, not being an inspector, wilfully alters, effaces, or obliterates, or causes to be altered, effaced or obliterated, wholly or partially, any mark on any article which has undergone inspection shall incur a penalty of one hundred dollars.

SEC. 19. *Appointment of officers.* The Minister may appoint inspectors and other officers for the carrying out of the provisions of this Act, but such appointments shall be confirmed by the Governor in Council within thirty days of the date thereof.

2. No person shall be appointed as a veterinary inspector until he has passed such examination as is deemed necessary by the Governor in Council.

SEC. 20. *Regulations.* The Governor in Council may make such orders and regulations, not inconsistent with the provisions of this Act, as to him seem necessary for the carrying out of the provisions of this Act.

2. Such orders and regulations shall have the same force and effect as if embodied in this Act.

3. Every such order or regulation shall be published twice in *The Canada Gazette*.

4. Any such order or regulation may be proved by the production of a copy thereof certified by the Minister; and such order or regulation shall, until the contrary is proved, be deemed to have been duly made and issued on the date thereof.

SEC. 21. *Inspector's certificate as evidence.* The certificate of the inspector or other officer appointed under the provisions of this Act shall, for the purpose of this Act, be prima facie evidence in all courts of justice and elsewhere of the matter certified.

SEC. 22. *Inspector's power of entry.* Any inspector or other officer appointed under the provisions of this Act may, at any time, for the purpose of carrying into effect any of the provisions of this Act, enter any place or premises, or

any steamship, vessel or boat, or any carriage, car, truck, horse-box or other vehicle used for the carriage of articles subject to the provisions of this Act, but shall, if required, state in writing the grounds on which he has so entered.

SEC. 23. *Obstructing inspector.* Every person who refuses to admit, or who obstructs or impedes, an inspector or other officer acting in execution of this Act, or of any order or regulation made by the Governor in Council or the Minister thereunder, and every person who aids and assists him therein, shall, for every such offence, incur a penalty not exceeding five hundred dollars; and the inspector or other officer may apprehend the offender and take him forthwith before a justice of the peace to be dealt with according to law; but no person so apprehended shall be detained in custody, without the order of the justice, longer than twenty-four hours.

SEC. 24. *Unlawful removal.* Every person who moves, or causes or allows to be moved, any animal, or any article in violation of the provisions of this Act, shall, for every such offence, incur a penalty not exceeding five hundred dollars.

SEC. 25. *Bribery of inspector.* The provisions of *The Criminal Code* respecting the bribery and corruption of officials or employees of the Government extend to all inspectors and other persons appointed to carry out the provisions of this Act.

SEC. 26. *Violations of Act.* Every person who violates any provision of this Act, or of any regulation made by the Governor in Council or by the Minister under the authority of this Act, in respect to which no penalty is hereinbefore provided, shall for every such offence, incur a penalty not exceeding five hundred dollars.

SEC. 27. *Apprehension of offenders.* Any inspector or constable may, without warrant, apprehend any person found committing an offense against the provisions of this Act, and shall take any person so apprehended forthwith before a justice of the peace to be examined and dealt with according to law; but a person so apprehended shall not be detained in custody, without the order of a justice, longer than twenty-four hours; and any inspector or constable may require that any animal or any article moved in violation of the provisions of this Act be forthwith taken back within the limits of the place whence it was moved, and may enforce and execute such requisition at the expense of the owner of such animal or article.

SEC. 28. *Place of committing of offence.* Every offence against this Act, or against any order or regulation of the Governor in Council or of the Minister, shall for the purpose of proceedings under this Act, or of any such order or regulation, be deemed to have been committed, and every cause of complaint under this Act, or any such order or regulation, shall be deemed to have arisen, either in the place in which it actually was committed or arose, or in any place in which the person charged or complained against happens to be.

SEC. 29. *Recovery of penalties.* Every penalty imposed by this Act shall be recoverable, with costs, before any two justices of the peace, or any magistrate having the powers of two justices of the peace, under Part XV. of *The Criminal Code*.—As amended June 16, 1908. 7-8 *Edward VII*, ch. 47.

SEC. 30. *Administration of Act.* The administration of any part of this Act may be assigned by the Governor in Council to any Minister other than the Minister of Agriculture, and in such case the Minister to whom such assignment is made shall have the same powers with respect to the part of this Act to him assigned as the Minister of Agriculture now has.

SEC. 31. *Suspension of operation.* The Governor in Council may suspend the operation of any of the sections of this Act until the first day of January next.

CONNECTICUT.

See Appendix, Bulletin 112, Part I, page 152, for general food laws, passed July 31, 1907, and included in that publication for convenience, though the compilation covered only laws passed in the fiscal year ended June 30, 1907.

KENTUCKY.

GENERAL FOOD LAWS.

SEC. 1. *Adulterated or misbranded food unlawful; penalty; proviso.* That it shall be unlawful for any person, persons, firm or corporation within this State to manufacture for sale, produce for sale, expose for sale, have in his or their possession for sale or to sell any article of food or drug which is adulterated or misbranded within the meaning of this act; and any person or persons, firm or corporation who shall manufacture for sale, expose for sale, have in his or their possession for sale or sell any article of food or drug which is adulterated or misbranded within the meaning of this act shall be fined not less than ten dollars nor more than one hundred dollars, or be imprisoned not to exceed fifty days or both such fine and imprisonment. Provided, That no article of food or drug shall be deemed misbranded or adulterated within the provisions of this act when intended for shipment to any other State or country, when such article is not adulterated or misbranded in conflict with the laws of the United States; but if said article shall be in fact sold or offered for sale for domestic use or consumption within this State, then this proviso shall not exempt said article from the operations of any of the other provisions of this act.

SEC. 2. *Food defined.* That the term food, as used in this act, shall include every article used for or entering into the composition of food or drink for man or domestic animals, including all liquors.

SEC. 3. *Misbranding defined.* For the purpose of this act, an article of food shall be deemed misbranded:

First. If the package or label shall bear any statement purporting to name any ingredient or substance as not being contained in such article, which statement shall not be true in any part; or any statement purporting to name the substances of which such article is made, which statement shall not give fully the name or names of all substances contained in any measurable quantity.

Second. If it is labeled or branded in imitation of or sold under the name of another article, or is an imitation either in package or label of another substance of a previously established name; or if it be labeled or branded so as to deceive or mislead the purchaser or consumer with respect to where the article was made or as to its true nature and substance, or as to any identifying term whatsoever whereby the purchaser or consumer might suppose the article to possess any property or degree of purity or quality which the article does not possess.

Third. If in the case of certified milk, it be sold as or labeled "certified milk," and it has not been so certified under rules and regulations by any county medical society, or if when so certified it is not up to that degree of purity and quality necessary for infant feeding.

Fourth. If it be misrepresented as to weight or measure or, if where the length of time the product has been ripened, aged or stored, or if where the length of time it has been kept in tin or other receptacle, tends to render the article unwholesome, the facts of such excessive storage, ripening, aging or packing are not plainly made known to the purchaser and to the consumer.

Fifth. If the package containing it or its label shall bear any statement, design, or device regarding the ingredients or the substances contained therein, which statement, design or device shall be false or misleading in any particular. Provided, That articles of liquor which do not contain any added poisonous or deleterious ingredients shall not be deemed to be adulterated or misbranded within the provisions of this act, in the case of articles labeled, branded or tagged so as to plainly indicate that they are compounds, imitations, or blends, and the word "compound," "imitation," or "blend," as the case may be, is plainly stated on the package in which it is offered for sale. Provided, That the term blend as used herein shall be construed to mean a mixture of like substances, not excluding harmless coloring and flavoring ingredients used for the purpose of coloring and flavoring only.

SEC. 4. *Adulteration defined.* For the purpose of this act, an article of food shall be deemed to be adulterated:

First. If any substance or substances be mixed or packed with it so as to reduce, lower or injuriously affect its quality or strength.

Second. If any substance be substituted in whole or in part for the article.

Third. If any valuable constituent of the article has been wholly or in part abstracted; or if the product is below that standard of quality represented to the purchaser or consumer.

Fourth. If it is mixed, colored, coated, polished, powdered, or stained whereby damage is concealed, or if it is made to appear better or of greater value than it is, or if it is colored or flavored in imitation of the genuine color or flavor of another substance of a previously established name.

Fifth. If it contains added poisonous ingredient which may render such article injurious to health, or if it contains any antiseptic or preservative which may render such article injurious to health, or any other antiseptic or preservative not evident or not plainly stated on the main label of the package.

Sixth. If it consists of or is manufactured from in whole or in part of a diseased, contaminated, filthy or decomposed substance, either animal or vegetable, unfit for food, or an animal or vegetable substance produced, stored, transported or kept in a condition that would render the article diseased, contaminated or unwholesome, or if it is any part the product of a diseased animal, or the product of an animal that has died otherwise than by slaughter, or that been^a fed upon the offal from a slaughterhouse, or if it is the milk from an animal fed upon a substance unfit for food for dairy animals or from an animal kept and milked in a filthy or a contaminated stable or in surroundings that would render the milk contaminated. Provided, That any article of food which may be adulterated and not misbranded within the meaning of this act, and which does not contain any added poisonous or deleterious ingredient and which is not otherwise adulterated within the meaning of paragraphs four, five and six of section four of this act, or which does not contain any filler or ingredient which debases without adding food value, can be manufactured or sold, if the same be labeled, branded or tagged so as to show the exact character thereof. And all such labels and all labeling of packages provided for in any provisions of this act shall be on the main label of each package and in such position and character of type and terms as will be plainly seen, read and understood by the purchaser or consumer. Provided further, That nothing in this act shall be construed as requiring or compelling the proprietors, manufacturers or sellers of proprietary foods which contain no unwholesome substances or ingredients to disclose their trade formulas except in so far as the provisions of this (act) require to secure freedom from adulteration, imita-

^a So in Statutes.

tion or misbranding. But in the case of baking powders, every can or other package shall be labeled so as to show clearly* the name of the acid salt which shall be plainly stated in the face of the label to show whether such salt is cream of tartar, phosphate or alum. Provided further, That nothing in this act shall be construed to prohibit the manufacture or sale of oleomargarine, butterine, or kindred compounds in a separate and distinct form, and in such manner as will advise the consumer of the real character, free from coloration or ingredient that causes it to look like butter.

[Sections 5 to 7, relate to drugs.]

SEC. 8. *Director of experiment station to make analysis, fix methods and standards; board for establishing regulations.* It shall be the duty of the Director of the Kentucky Agricultural Experiment Station, or under his direction, the head of the division of food inspection of the said station, to make or cause to be made examinations of samples of food and drugs manufactured or on sale in Kentucky at such time and place and to such extent as he may determine. He shall also make, or cause to be made, analysis of any sample of food or drug which the State Board of Health or the State Board of Pharmacy may suspect of being adulterated or misbranded, and of any sample of food or drug furnished by any Commonwealth's, county or city attorney of this State. And the said director may appoint such agent or agents as he may deem necessary, who shall have free access at all reasonable hours for the purpose of examining into places wherein any food or drug product is being produced, manufactured, prepared, kept or offered for sale, for the purpose of determining as to whether or not any of the provisions of this act are being violated, and such agent or agents upon tendering the market price of any article may take from any person, firm or other corporation, a sample of any article desired for examination.

The director of said Experiment Station is hereby empowered to adopt and fix the methods by which the samples taken under the provisions of this act shall be analyzed or examined, and to adopt and fix standards of purity, quality or strength, when such standards are necessary or are not specified or fixed herein or by statute. Provided, That such standards shall be published for the information and guidance of the trade. Provided further, That for the purpose of uniformity, when such standards so fixed differ from the legally adopted standards of the United States Department of Agriculture, the director of said station shall arrange for a conference between the proper food control representatives of the United States Department of Agriculture and the director of said station and the representatives of the trade to be affected, for the purpose of arriving, if possible, at a uniform state and national standard. Provided further, That in the case of final dispute the validity of such standards adopted by the director of said station shall be determined by the Courts under the rules of evidence. And Provided further, That when the standard or nomenclature for any food or food product has been determined by the Supreme Court of the United States such standard or nomenclature shall govern in the enforcement of the provisions of this act. Provided further, That all rulings pertaining to sanitation under this act shall be collaborated in connection with the State Board of Health. And provided further, That at the regular annual meetings of the Kentucky Pharmaceutical association and the Kentucky State Medical association each of said associations shall elect one representative, which representatives, together with the director of said station shall make and establish all rules and regulations for the governing and carrying out of the provisions of this act relating to drugs.

* So in Statutes.

Sec. 9. Prosecution. Whenever any article shall have been examined and found to be adulterated or misbranded in violation of this act, the Director shall certify the facts to the Commonwealth's attorney of the district, or to the county attorney of the county, or the city attorney of any city or town, in which the said adulterated or misbranded food or drug product was found, together with a statement of the results of the examination of said article of food or drug, duly authenticated by the analyst under oath and taken before some officer of this Commonwealth authorized to administer an oath having a seal. And it shall be the duty of every Commonwealth's attorney, county attorney and city attorney to whom the Director of said station shall report any violation of this act or to whom the State Board of Health, or the State Board of Pharmacy, or to whom the chief health officer of any county, city or town shall report any such violations, to cause proceedings to be commenced against the party so violating the act, and the same prosecuted in manner as required by law. Provided, however, That in case of the first charge or finding the manufacturer or dealer shall be notified of the findings and be given a hearing within fifteen days before a report is made to the Commonwealth's, county or city attorney as herein provided. Provided further, That where more than one sample of the same brand of product has been taken and examined, the first finding or charge shall be construed to apply to all samples so taken, and notice and hearing shall apply to all such samples.

Sec. 10. Annual report; provisos. Said station shall make an annual report to the Governor upon adulterated food or drug products in addition to the reports required by law which shall not exceed one hundred and fifty pages, and such annual reports shall be submitted to the General Assembly at its regular session, and said station may issue from time to time a bulletin giving the results of the inspections and of all analyses of samples taken or submitted for examination under this act, together with the names of the parties from whom the samples were taken, or where the inspections were made, and as far as possible the name of the manufacturers, the number of samples found to be adulterated, the number found not adulterated, and other information which may be of interest to the manufacturers or dealers in food or drug products or to the consumers. Provided, however, That before such publication is made the manufacturer of the article and the dealer shall be furnished a true copy of the facts to be published regarding the article at least thirty days before the publication and hearing given the dealer and manufacturer, and any statements or explanations made by such manufacturer shall be included in the same place and along with the publication made regarding the article. And provided further, That if at the hearing of the manufacturer or dealer, as provided by section 9 hereof, said manufacturer shall produce the affidavit of a competent analytical chemist controverting the finding of said station or its director or chemist, as the case may be, and affirmatively showing that there is neither adulteration or misbranding of such article under the provisions of this act, then there shall be no publication of either the name of the manufacturer or dealer, or of the name of the brand of the article until after a trial and a verdict of guilty as herein provided. And provided further, That where prosecution is made for violation of any of the provisions of this act, no official publication shall be made of the result of the inspection and analysis until the matter has been finally adjudicated, and in case of appeal, by the court of last resort.

Sec. 11. Cost of analysis; appropriation; expenditures. Said Experiment Station shall receive seven dollars and fifty cents (\$7.50) for the analysis or examination of any sample of food or drug taken or submitted in accordance with

^a So in Statutes.

this act, and expenses for procuring samples of food and drugs and in making inspections into the condition of and wholesomeness and purity of the food produced, manufactured or sold in food factories, grocery stores, bakeries, slaughtering houses, dairies, milk depots or creameries, and all other places where foods are produced, prepared, stored, kept or offered for sale; for studying the problems connected with the production, preparation and sale of foods; for expert witnesses attending grand juries and courts; clerk hire and all other expenses necessary for carrying out the provisions of this act. Provided, The total expense from all sources shall not exceed in any one year thirty thousand dollars (\$30,000.00.)

The Board of Control of said Experiment Station shall furnish to the Auditor of Public Accounts an itemized statement of the expenditures of money under this act. The expenditures reported to the Auditor shall be paid by the Commonwealth to the treasurer of the Experiment Station upon the written request of the Board of Control of the said Experiment Station, and the Auditor for the payment of the same is directed to draw his warrant upon the Treasurer as in all other claims against the Commonwealth.

SEC. 12. *Filing of label, brand, etc.* When any manufacturer shall offer any article of food or drug for sale in the State he shall file with the director of the said station, when requested by him, the name of the brand, the name of the product, the place of its manufacture or preparation, and a true copy of all labeling used thereupon. Failure to so file within thirty days shall be punished as provided in section 1 of this act.

SEC. 13. *Guaranty as evidence.* In all prosecutions under this act, the courts shall admit as evidence a guaranty which has been made to the holder of the guaranty by any manufacturer or wholesaler residing in this State, to the effect that the product complained of is not adulterated or misbranded within the provisions of this act. And said guaranty, properly signed by the wholesaler, jobber or manufacturer or other party residing within this State from whom the holder of the guaranty may have purchased the article or articles complained of, and containing the full name and address of the party or parties making the sale of such article to the holder of the guaranty, and in the absence of any proof that the article or articles complained of were adulterated or misbranded after they had been received by the holder of the guaranty, shall be a bar to prosecution of the holder of such guaranty under the provisions of this act.

SEC. 14. *Repeal.* All acts or parts of acts inconsistent herewith are hereby repealed, but this said act shall not be construed to repeal Chapter 48 of the Acts of the General Assembly of 1906, entitled, "An Act to regulate the sale of concentrated commercial feeding stuffs, defining same and fixing penalties for violations thereof."

So much of this act as relates to drugs and liquors shall not take effect until on and after January 1, 1909.

Approved March 13, 1908. Acts of 1908, ch. 4, pp. 10-22.

DAIRY PRODUCTS.

See General Food Laws.

LIQUORS.

See General Food Laws.

LOUISIANA.

The food and drug regulations of the Louisiana board of health have the force of law and are therefore quoted in full except when they are the same as the Federal law, regulation, or standard on a given point, in which case the appropriate section is referred to.

REGULATIONS.

REG. 1. *Title of the Rules and Regulations.* The Louisiana State Board of Health hereby decrees and establishes the following Rules and Regulations governing the manufacture, sale, and inspection of foods, liquors, waters, and drugs within the State.

The Rules and Regulations of the Louisiana State Board of Health governing the manufacture, sale and inspection of foods, drugs, liquors or waters, shall be known and referred to as "The Food and Drug Regulations of the Louisiana State Board of Health."

REG. 2. *Prohibiting adulteration or misbranding of food and drugs.* It shall be unlawful for any person or persons to manufacture within this State any article of food, drugs, liquors or waters which is adulterated or misbranded within the meaning of these Regulations; and any person who shall violate any of the provisions of these Regulations shall be punished as provided for in Sec. 3, Act 98 of 1906.

REG. 3. *Prohibiting importation or exportation of adulterated or misbranded food, drugs, liquors or waters.* That the introduction into this State from any other State or Territory, or from the District of Columbia, or from any foreign country, of any article of food, drugs, liquors or waters, which is adulterated or misbranded within the meaning of these Regulations is hereby prohibited, that any person who shall receive from any State or Territory, or the District of Columbia or foreign country, and having so received, shall deliver in unbroken or broken packages, for pay or otherwise, or offer to deliver to any other person any such article so adulterated or misbranded within the meaning of these Regulations, or any person who shall sell or offer for sale, or have in his possession for sale in this State any such adulterated or misbranded foods, drugs, liquors or waters, shall be punished as provided for in Section 3, Act 98 of 1906.

Provided: That no article shall be deemed misbranded or adulterated within the provisions of these Regulations, when intended for export to any foreign country and prepared and packed according to the specifications or directions of the foreign purchaser when no substance is used in the preparation or packing thereof in conflict with the laws of the foreign country to which said article is intended to be shipped; but if said article shall be in fact sold or offered for sale for domestic use or consumption, then this provision shall not exempt said article from the operation of any of the other provisions of these Regulations.

Broken or unbroken packages defined. The term "Broken or unbroken package" as used in these Regulations, is the original package or part thereof, carton, case, box, barrel, bottle, phial, or other receptacle put up by the manufacturer to which the label is attached, or which may be suitable for the attachment of a label, making one complete package of the food, drug, liquor or water article.

The original package contemplated includes both the wholesale and the retail packages.

REG. 4. State Food Commissioner, Dairy Commissioner, Analyst. The President of the Louisiana State Board of Health shall be ex officio State Food Commissioner. The State Food Commissioner may, with the advice and consent of the Louisiana State Board of Health, appoint two or more Assistant Commissioners, each one of acknowledged standing, ability, and integrity, one of whom shall be an expert in the matter of dairy products, and one of them shall be a practical and Analytical Chemist, who shall be known as the State Analyst. The salaries of each assistant shall be fixed by the State Food Commissioner, by and with the consent of the Louisiana State Board of Health. In case of the absence or inability of the State Analyst to perform all the duties of his office or for the purpose of expediting the work of the Department, the State Food Commissioner may appoint some competent person to assist in the same temporarily.

REG. 5. Inspectors. The State Food Commissioner shall have authority, by and with the consent of the Louisiana State Board of Health, to appoint necessary inspectors, to assist in the work of the State Food Commissioner at such times and for such periods of time as may be required in the enforcement of the dairy, drug and food laws of the State. Such Inspectors shall have the same right of access to places to be inspected as the State Food Commissioner. The compensation of such Inspectors shall be fixed by the State Food Commissioner, by and with the consent of the Louisiana State Board of Health.

REG. 6. Duty of State Food Commissioner. It shall be the duty of the State Food Commissioner to enforce all the rules and regulations herein provided for or that may hereafter be enacted by this Board regarding the production, manufacture or sale of dairy products or the adulteration of any article of food or drugs, liquors or waters, and personally or through his assistants, to inspect any article of food, drugs, liquors or waters, made or offered for sale or held in possession for sale, which he may, through himself or his assistants, expect or have reason to believe to be impure, unhealthful, adulterated or misbranded, and to prosecute or cause to be prosecuted any person or persons, firm or firms, corporation or corporations, engaged in the manufacture or sale of any adulterated or misbranded article or articles of food, drugs, liquors or waters, contrary to these Regulations.

REG. 7. Examination of foods, drugs, liquors or waters, collection of samples. Methods of analysis. The examination of foods, drugs, liquors or waters shall be made by the State Analyst or his Assistants under the direction of the State Food Commissioner for the purpose of determining from such examinations whether such articles are adulterated or misbranded within the meaning of these regulations, and if it shall appear from any such examination that any of such specimens are adulterated or misbranded within the meaning of these Regulations, the State Food Commissioner shall cause notice thereof to be given to the party from whom such sample was obtained; any party so notified shall be given an opportunity to be heard under such other rules and regulations as may be prescribed by this Board, and if it appears that any of their rules and regulations have been violated by such party, then the State Food Commissioner shall at once certify the facts to the District Attorney of the District

wherein the offense was committed with a copy of the results of the analysis or the examination of such article duly authenticated by the Analyst or officer making such examination under the oath of such officer: after judgment of the Court, notice shall be given by publication in such manner as may be prescribed by this Board.

REG. 8. *Collection of samples.* Samples of broken or unbroken packages shall be collected only by Inspectors appointed by the State Food Commissioner, or by the Health, Food, or Drug Officer of the cities and towns of Louisiana. Samples may be purchased in the open market, and if in bulk, the marks, brands or tags upon the package, carton, container, wrapper or accompanying printed or written matter shall be noted. The Inspector shall also note the names of the vendor and agent through whom the sale was actually made, together with the date of purchase. The Inspector shall purchase representative samples. A sample taken from bulk goods shall be divided into three (3) parts and each part shall be labeled with the identifying marks. All samples shall be sealed by the Inspector with a seal provided for the purpose. If the package be less than four (4) pounds, or in volume less than two (2) quarts, three (3) packages of approximately the same size shall be purchased when practicable, and the marks and tags upon each noted as above. One sample shall be delivered to the party from whom purchased, one sample shall be sent to the Food Laboratory of the State Analyst, and the third sample shall be held under seal by the State Food Commissioner.

REG. 9. *Methods of analysis.* Unless otherwise directed by the State Board of Health the methods of analysis employed shall be those prescribed by the Association of Official Agricultural Chemists and the United States Pharmacopœia.

REG. 10. *Hearings.* (A) When the examination or analysis shows that the provisions of the "Food and Drug Regulations of the Louisiana State Board of Health" have been violated, notice of the fact together with a copy of the findings shall be furnished to the party or parties from whom the sample was obtained, and a date shall be fixed at which such party or parties may be heard before the State Food Commissioner, or such other official connected with the Food and Drug Inspection service as may be commissioned by the State Food Commissioner for that purpose; the hearings shall be held at a place to be designated by the State Food Commissioner most convenient for all parties concerned. These hearings shall be private and confined to questions of fact. The parties interested therein may appear in person, or by Attorney, and may propound interrogatories, and submit oral or written evidence to show any fault or error in the findings of the Analyst or Examiner. The State Food Commissioner may order a re-examination of the samples or have new samples drawn for further examination.

When an article examined by the State Analyst is found to come in conflict with the regulations of the Louisiana State Board of Health, a written notice shall be served at once on the person or persons, or dealer or dealers, offering the same for sale, warning him or them not to sell or expose for sale such condemned article or articles.

(B) Whenever it would appear to the best interest of the public health and welfare, the Food Commissioner of the Louisiana State Board of Health is required to render such condemned articles of food, drugs, liquors or waters, unfit for consumption by man or animals.

(C) In the event that such person or persons, shall continue to violate these Regulations by selling, offering for sale, or hold in possession for sale or barter, such condemned article or articles, the State Food Commissioner shall lay

before the District Attorney of the District in which the violation occurred, the evidence of such violation, together with a copy of the analysis of the State Analyst.

(D) In the event the District Attorney should fail to promptly institute proceedings in a court of competent jurisdiction, the State Food Commissioner shall place the whole matter in the hands of the Attorney General of the State.

REG. 11. *Definition of the words foods and drugs as used herein.* (A) The term drug as used in these Regulations shall include all substances, compounds, and preparations recognized in the United States Pharmacopœia or National Formulary, for internal or external use, and any other substance or mixture of substances intended to be used for the cure, mitigation or prevention of disease of either man or other animals.

(B) The term "food" as used herein, shall include all articles intended for food, drink, confectionery, condiment, or used in the preparation thereof, whether simple, mixed, or compound.

REG. 12. *Food adulterations defined.* For the purposes of these Regulations, an article shall be deemed to be adulterated in case of foods: See Standards for Foods, Reg. No. 45; [also the Federal Food and Drugs Act, "Sec. 7. In the case of food," 1 to 6, inclusive, with the addition of the following clause after "Fifth"]: Not excluded under this provision are substances properly used in the preparation of food products for clarification or refining, and elimination in the further process of manufacture.

Powdering, coating, and staining. [See Federal Reg. 12.]

REG. 13. *Misbranding.* [See Federal Food and Drugs Act, sec. 8, also.]

In case of drugs: * * *

Fourth—If the package containing it or its label shall bear any statement, design or device which shall be false or misleading in any particular.

Provided: That an article of food which does not contain any added poisonous or deleterious ingredients shall not be deemed to be adulterated or misbranded in the following cases:

First—In the case of mixture or compounds which may be now or from time to time hereafter known as articles of food, under their own distinctive names, and not an imitation of or offered for sale under the distinctive name of another article, if the name be accompanied on the same label or brand with a statement of the place where said article has been manufactured or produced.

Second—In the case of articles labeled, branded or tagged so as to plainly indicate that they are compounds, imitations, or blends, and the word "compound," "imitation," or "blend" as the case may be, is plainly stated on the package in which it is offered for sale. Provided: That the term blend, as used herein, shall be construed to mean a mixture of like substances.

REG. 14. *Label.* (a) The term "label" applies to any printed, written, pictorial, or other matter upon or attached to any package of a food or drug product, or any container thereof, including ink written, typewritten, or stencilled labels of druggists.

(b) The principal label shall consist, first—the name of the substance or product; the name of place of manufacture in the case of food compounds or mixtures; words which show that the articles are compounds, mixtures or blends; the words "compound," "mixture" or "blend," or the words designating the substances or their derivatives, and proportions required to be named in the case of drugs; and in the case of foods, the constituents are to be named in the order of their relative proportion.

All these required words shall appear upon the principal label with no intervening description or explanatory reading matter.

Second—If the name of the manufacturer and place of manufacture are given, they shall also appear upon the principal label.

Third—Elsewhere upon the principal label other matter may appear in the description of the manufacturer.

(c) The principal label on food or drugs for domestic commerce shall be printed in English (except as hereinafter provided for), with or without the foreign label in the language of the country where the food or drug product is produced or manufactured.

The size of type shall not be smaller than 8-point (brevier) caps: Provided, that in case the size of the package will not permit the use of 8-point (brevier) cap type, the size of the type may be reduced proportionately.

(d) The form, character and appearance of the labels, except as provided above, are left to the judgment of the manufacturer.

(e) Descriptive matter upon the label shall be free from any statement, design or device regarding the article or the ingredients or substances contained therein, or quality thereof or place of origin, which is false or misleading in any particular.

(f) An article containing more than one food product or active medicinal agent, is misbranded if named after a single constituent.

(g) The term "design" or "device" applies to pictorial matters of every description, and to abbreviations, characters, or signs for weights, measures or names of substances.

(h) The use of any false or misleading statement, design or device shall not be justified by any statement given as the opinion of an expert or other person, appearing on any part of the label, nor by any descriptive matter explaining the use of the false or misleading statement, design or device.

Name and address of manufacturer. [See Federal Reg. 18-20 and secs. (b) and (c) of Reg. 21.]

Distinctive name. [See Federal Reg. 20.]

(u) A color or flavor cannot be employed to imitate any natural product or any other product of recognized name and quality, except as especially provided for in Regulations 38 and 45—sections covering Root Beer, Candy and Confectionery.

Imitation. [See Federal Regs. 21-22.]

Reg. 15. *Mixtures or compounds, with distinctive names.* [See Federal Reg. 27.]

Reg. 16. *Proper branding not a complete guarantee.* [See Federal Reg. 23.]

Reg. 17. *Incompleteness of branding.* [See Federal Reg. 24.]

Reg. 18. *Substitution.* [See Federal Reg. 25.]

Reg. 19. *Waste material.* [See Federal Reg. 26.]

Reg. 20. [Relates to drugs.]

Reg. 21. *Inspection of raw materials.* The State Food Commissioner, when he deems it necessary, shall examine or cause to be examined, the raw materials used in the manufacture of food and drug products, and determine whether any filthy, decomposed, or putrid substance is used in their preparation.

Reg. 22. *Working formula required.* The State Food Commissioner shall have furnished him on demand a certified copy of the working formula used in the manufacture of any compound of drugs, foods, liquors or waters, when in his judgment the safety of the public health and the enforcement of these Regulations demand it. It being well understood that said certified copy be and remain the property of the manufacturer furnishing the same; and shall be regarded strictly as a confidential communication.

Reg. 23-25. [Relate to drugs.]

REG. 26. *Substances named in drugs or foods.* [See Federal Reg. 28, a, b, c, and f.]

The following articles shall be included in the above list and become subject to the rules and regulations governing it on and after October 1, 1908:

Nux vomica, its active principles and preparations.
 Gelsemium, its active principles and preparations.
 Physostigma, its active principles and preparations.
 Belladonna, its active principles and preparations.
 Scopola, its active principles and preparations.
 Hyoscyamus, its active principles and preparations.
 Stramonium, its active principles and preparations.
 Veratrum viride, its active principles and preparations.
 Staphisagria, its active principles and preparations.
 Aconite, its active principles and preparations.
 Colchicum, its active principles and preparations.
 Pilocarpus, its active principles and preparations.
 Pelletierine, its active principles and preparations.
 Conium, its active principles and preparations.
 Scopolius, its active principles and preparations.
 Digitalis, its active principles and preparations.
 Convallaria, its active principles and preparations.
 Strophanthus, its active principles and preparations.
 Male fern, its active principles and preparations.
 Santonin, its active principles and preparations.
 Ergot, its active principles and preparations.
 Gossypii cortex, its active principles and preparations.
 Elaterium, its active principles and preparations.
 Croton oil, its active principles and preparations.
 Cantharides, its active principles and preparations.
 Antimony, its compounds and preparations.

Mercury, its compounds and preparations, except calomel and mercury in metallic state.

Arsenic, its compounds and preparations.

Potassium cyanide and hydrocyanic acid.

Carbolic acid.

Any synthetical compound having the property of relieving pain, producing sleep, or reducing temperature.

REG. 27. [*Relates to drugs.*]

REG. 28. *Statement of weight or measure.* [See Federal Reg. 29, also the following]:

(c) In the case of alcohol the expression "quantity" or "proportion" shall mean the average percentage by volume in the finished product.

(d) In the case of the other ingredients required to be named upon the label, the expression "quantity" or "proportion" shall mean grains or minims per ounce or fluid ounce, per unit, per tablet, pill, etc., and also, if desired, the metric equivalents therefor, or milligrams per gram or per cubic centimeter, or grams or cubic centimeters per kilogram or per litre.

REG. 29. *Imported food and drug products.* Food products intended for export containing added substances not permitted in foods intended for consumption in this State, but in accordance with the directions of the foreign purchaser, must be kept separate and labeled to indicate that they are for export.

If these products are not exported, they shall not be allowed to be sold, bartered or given away for consumption in this State.

Meat and meat food products as well as all other food and drug products of a kind forbidden entry into or forbidden to be sold, or restricted in sale in the country in which made or from which exported, must not be sold, bartered or given away in this State.

REG. 30. *Denaturing*. [See Federal Reg. 34.]

REG. 31. *Instruction to inspectors*. In sending in samples for analysis to this Department of any manufactured product, the following information must accompany each sample, to-wit:

(a) Name and location of manufacturer or dealer. If bought of jobbers, the firm name and location plainly written in ink.

Brand or name of article, any representation by seller as to quality or character of goods.

To enable correct analysis to be made, not less than the following quantities of each article should be sent:

Bread, not less than 16 ounces.

Butter, not less than 8 ounces.

Baking powder, not less than 1 small can.

Beer, not less than 1 pint.

Buckwheat flour, not less than 8 ounces.

Cheese, not less than 6 ounces.

Candy, not less than 8 ounces.

Cocoa and

Chocolate, in small original package.

Cream of tartar, not less than 1 ounce.

Cream, not less than 4 ounces.

Extracts, not less than 2 ounces.

Honey, not less than 8 ounces.

Jellies, not less than $\frac{1}{2}$ lb., or small original package.

Jams, not less than $\frac{1}{2}$ lb., or small original package.

Liquor, not less than 1 pint.

Lard, not less than 4 ounces.

Maple sugar, not less than 1 pound.

Molasses and syrups, not less than 1 pint.

Milk, not less than 4 ounces.

Olive oil, not less than 4 ounces.

Preserves, not less than $\frac{1}{2}$ lb., or small original package.

Spices, not less than 4 ounces.

Sugar, not less than 8 ounces.

Vinegar, not less than 1 pint.

Wine, not less than 1 pint.

Goods should be procured in original package when put up in packages containing not more than two pounds solid or one-half gallon liquid measure.

REG. 32. *Baking powder*. No person in this State shall make or manufacture baking powder or any other mixture or compound intended for use as baking powder, or sell, exchange, deliver, or offer for sale or exchange, such baking powder, or any mixture or compound intended for use as baking powder, unless its composition be distinctly shown by a label on the outside and face of which is printed with black ink in a legible type, with roman letters not less than 8-point—brevier—cap on a white or light black background, the manufacturer's name and the place of manufacture and in a conspicuous place on the face of the label of such package of baking powder and with letters similar in size, the name of the acid ingredient together with a list of all the ingredients entering into its composition.

Provided, the use of any substance deemed poisonous or injurious is hereby prohibited and the use thereof in the manufacture of baking powder is hereby declared unlawful. Baking powders must yield at least 8 per cent available carbon dioxide. The use of argolite, terra alba and all other mineral fillers is prohibited.

Baking powders must have specific name of the powder on the label and no ingredient can be named as a component of the powder not found in the article.

REG. 33. *To regulate the manufacture and sale of substitutes of butter.* (a) That for the purpose of these regulations every article, substitute or compound of any other than that which is produced from pure milk or cream, therefrom made in a semblance of butter and designated to be used as a substitute for butter made from pure milk or its cream, is hereby declared to be imitation butter.

Provided: That the use of salt and harmless coloring matter for coloring the product of pure milk or cream shall not be construed to render such product an imitation.

(b) No person shall coat, powder or color with annato or any injurious coloring matter whatever, any substance designed as a substitute for butter whereby such substitute or product so colored or compounded shall be made to resemble butter, the product of the dairy and sold as such. No person shall combine any animal fat with butter and sell the same for consumption.

Provided: nothing in these regulations shall be construed to prohibit the use of salt, rennet and harmless coloring matter for coloring the product of pure milk or cream from the same.

(c) No person shall produce or manufacture any substance or semblance in imitation of natural butter, nor sell or keep for sale, barter or give away, nor offer for sale any imitation butter made, manufactured, compounded or produced in violation of this Regulation whether such imitation butter shall be made or produced in this State or elsewhere; this Regulation shall not be construed to prohibit the manufacture and sale under the Regulations hereinafter provided of substances designed to be used as a substitute for butter, and not manufactured or colored as herein provided.

(d) Every person who lawfully manufactures any substance designed to be used as a substitute for butter, shall mark by branding, stamping or stenciling upon the top side of each box, tub, firkin, or other package in which such article shall be kept, and in which it shall be removed from the place where it is produced, in a clear and durable manner in the English language the word "Oleomargarine" or the word "Butterine" or the words "Substitute for Butter" or the words "Imitation Butter" in printed letters, in plain roman type; each of which shall not be less than three-fourths of an inch in length.

(e) It shall be unlawful to sell or offer for sale, barter or give away, any imitation butter without informing the purchaser thereof, or the person or persons to whom the same is offered for sale, that the substance sold or offered for sale is imitation butter.

(f) No person by himself or with others shall ship, consign or forward, by any common carrier whether public or private, any substance designed to be used as a substitute for butter, unless it shall be marked or branded on each tub, box, firkin, jar or other package containing the same, as provided in this Regulation, and unless it be consigned by the carriers and receipted for by its true name.

(g) No person shall have in his possession or under his control, any substance designed to be used as a substitute for butter unless the tub, firkin, jar, box or other package containing the same be clearly and durably marked as provided in this Regulation.

Every person who shall have possession or control of any imitation butter for the purpose of selling, bartering, or giving away the same, which is not marked as required by the provisions of this Regulation, shall be presumed to have known during the time of such possession or control, the true character and name as fixed by this Regulation of such product.

(h) Whoever shall have possession or control of any imitation butter, or any substance designed to be used as a substitute for butter contrary to the provisions of this Regulation, for the purpose of selling the same or offering the same for sale, barter or give away, shall be held to have possession of such property with intent to use it in violation of this Regulation.

(i) Whoever shall deface, erase or remove any mark provided by this Regulation, with intent to mislead, deceive or violate any of the provisions of this Regulation, shall be held liable to the penalties herein provided for a violation of any of these Regulations.

(j) That no person, firm, corporation, agent, or employe shall manufacture, sell, offer or expose for sale in this State, any butter that is produced by taking original packing stock butter, or other butter, or both, and melting the same, so that the butter fat can be drawn off or extracted, then mixing the said butter fat with skimmed milk, or milk, or cream, or other milk product and re-churning or reworking the said mixture, or that produced by any process that is commonly known as boiled, process, or renovated butter unless the same is branded or marked as provided in this Regulation.

(k) No person, firm, corporation, agent or employe shall sell, offer or expose for sale, barter, or give away, or deliver to purchaser any boiled, process, or renovated butter unless the words: "Renovated Butter" shall be plainly branded with gothic or bold faced letters at least three-fourths of an inch in length on the top and sides of each tub, or box, or pail, or other kind of a case or package, or on the wrapper of prints or rolls in which it is put. If such butter is exposed for sale uncovered or not in a case or package, a placard containing the label so printed, shall be attached to them in such a manner as to be easily seen and read by the purchaser. The branding or marking of all packages shall be in the English language, in a conspicuous place so as to be easily seen and read by the purchaser.

(l) Every hotel, restaurant or boarding house, using any imitation, processed, or renovated butter, must state the true nature of the imitation or processed butter used on the bill of fare or on a placard conspicuously placed, and printed in bold type and in the English language.

(m) The State Food Commissioner and his assistants, experts, chemists or agents shall have access and ingress to all places of business, factories, stores, and buildings used for the manufacture or sale of butter. They shall have power and authority to open any tub, box, pail or other kind of case or package containing any butter that may be manufactured, sold or exposed for sale.

REG. 34. Candy, confectionery, cocoa, chocolate. (a) In the case of confectionery:

It shall be considered adulterated if it contains terra alba, barytes talc, chrome yellow, or other mineral substance or poisonous color or flavor or other ingredient deleterious or detrimental to health, or any vinous, malt or spiritous liquor or compound or narcotic drug.

Candy must not be wrapped in tin foil in direct contact with the candy.

REG. 35. Canned goods. No packer or dealer in preserved or canned fruits and vegetables or other articles of food shall sell or offer for sale such canned or preserved fruits and vegetables or other articles unless they shall be entirely free from substances or ingredients deleterious to health, or use dyes

or coloring matter whereby their true character would be disguised or inferiority concealed.

The addition of sugar to a substance not naturally sweet, converting it into a substance which might seem naturally sweet is permitted, if the label plainly indicates that sweetening material has been added.

All soaked or bleached goods or goods put up from products dried before canning shall be plainly marked, branded, stamped or labeled as such with the words "Soaked" or "Bleached goods" in letters of equal size to that of the name of the product and bearing the name of the article and name and address of the packer or dealer who sells same.

REG. 36. *Cold storage.* The sale of milk or cream that has been kept over 24 hours in cold storage, the sale of fish that has been kept over 24 hours in cold storage, the sale of meat that has been kept over three weeks in cold storage is prohibited unless the facts in regard to the same are certified to the purchaser.

REG. 37. *Coffee.* Coffee must be true to name.

It must not be coated, colored or polished when such coating, coloring or polishing injures the coffee, or conceals some damage or inferiority.

Imitations containing no coffee cannot be sold as coffee compounds, but must be sold as imitation coffee, with the statement that they contain no coffee.

Compounds of coffee and chicory, or of coffee and any other harmless substitute allied to it, either in flavor or strength and not used simply as an adulterant may be sold when labeled Coffee Compound and such compound must state on face of label the names of the ingredients used in making the compound in the order of their relative proportions, in type of equal size and prominence.

REG. 38. *Fruit syrup, soda water syrup, pop, soft drinks, etc.* Drinks containing less than two per cent of alcohol, fruit syrups, soda water syrups, pops, soft drinks, etc., shall not contain any saccharin, salicylic, or boric acid, their derivatives, or any harmful coloring matter or preservative. All drinks containing less than two per cent of alcohol, fruit syrups, soda water syrups, pops, soft drinks, etc., made from any substance except the natural extract of fruit, or flavored or colored with synthetical products, must have the word "artificial" printed on the label of the package in the same size, style and color of letter and background as the name of the article, and in such a manner as to show that they have no relation whatsoever to the fruit which they imitate. All soda fountains or places where the above mentioned "artificial" articles are sold or served, shall have printed on a placard the words, "artificial drinks" and hung in front of the fountain or in a conspicuous place.

See Reg. 45 for list of permitted "Coal Tar Dyes."

The use of 1-10 of one per cent of benzoate of soda, is permitted in natural or artificial fruit syrups.

The use of saccharin in any food product is prohibited.

The terms "Artificial" and "Imitation" may be used synonymously.

REG. 39. *Honey.* No person, firm or corporation shall offer for sale, or possess with intent to sell, barter or give away, honey manufactured from or mixed with glucose, sugar, or syrup of any kind, or any substance not the legitimate product of the honey bee, unless the package containing same is so marked and represented as such and bearing a label upon the package printed in heavy Gothic capitals, 18 point, with the name of the person manufacturing or mixing the same, and the name of the substance or material from which it is compounded.

REG. 40. *Ice.* No person, firm or corporation shall manufacture, sell or deliver for food or drink purposes, any ice natural or manufactured, containing decomposed, putrid, infected, tainted, or rotten animal or vegetable substances,

or any ingredient injurious to health. 'Nor ice made from water of a lower standard of purity than that required for potable water by the State Board of Health, as indicated in its Standards and definitions of Food Products.

REG. 41. Lard. (a) No person, firm or corporation shall manufacture or sell lard to which has been added beef or mutton fat, stearine, cotton seed oil, or other substitute for swine fat, unless the container is plainly marked "adulterated" or "Lard Compound" in bold letters and the quantity and name of the adulterant is made part of the label.

(b) Lard, lard compounds, or lard substitutes, containing more than one (1) per cent of water, shall be considered adulterated.

REG. 42. Adulteration of wines. (a) All wine containing alcohol, except such as have been produced by natural fermentation of pure undried fruit juices, or combined with distilled spirits, whether denominated wines or by any other name, which may be used as a beverage or combined with other liquors intended for use, and all compounds of the same with pure wine, and all preserved fruit juices compounded with substances not produced from undried fruit intended for use as a beverage or for use in the fermentation or preparation of liquors intended for such use, and all wines, imitations of wines, or other beverages produced from fruit which shall contain alum, baryta, lime, carbonate of soda, salts of lead, salicylic acid, or any other antiseptic or coloring matter not produced from undried fruit, or which contains artificial flavoring, essence of ether or any other foreign substance injurious to health, shall be known as, or deemed to be adulterated wine, and shall not be sold, offered for sale, barter or give away or manufactured with intent to sell, barter or give away, within the State.

REG. 43. Sugars, syrups and molasses must conform to the standards laid down. (A) It shall be unlawful for any person or persons, firm or corporation or agent thereof, to sell, advertise, or offer for sale, barter or give away within the limits of this State, any compound or mixed syrup, unless at the time of sale, the names of the ingredients in the order of their relative proportion of such mixture or compound are clearly stamped or labeled on the bottle, can, case, barrel or other receptacle containing such syrup.

The term "Mixture or Compound" as used in this Regulation is understood to apply to all mixtures or compounds of two or more ingredients differing in their nature or quality such as sugar cane syrup, sorghum cane syrup, maple syrup, molasses or glucose (corn syrup.)

Finished syrups or molasses, containing zinc or tin compounds, will be condemned as food products.

All packages of mixed or compound syrups in barrels, cans, bottles or other containers shall be labeled with the name of the manufacturer and the place of manufacture.

(B) In the manufacture of syrups and molasses the use of sulphur as a clarifying agent is permissible, Provided: the residual sulphur does not exceed 1-10 of one per cent.

REG. 44. Principles on which the standards are based. [See Cir. 19, Office of the Secretary, Federal Standards. These were adopted in toto and only the standards adopted in addition to those promulgated by the Secretary of Agriculture (Cir. 19) are here given.]

REG. 45. Food standards. [See also Cir. 19, Office of the Secretary, Federal Standards.]

c. Meat extracts, meat peptones, gelatine, etc. 1. *Meat extract* is the product obtained by extracting fresh meat with boiling water and concentrating the liquid portion by evaporation after the removal of fat, and contains not less than seventy-five (75) per cent of total solids, of which not over twenty-seven

(27) per cent is ash, and not over twelve (12) per cent is sodium chlorid (calculated from the total chlorin present), not over six-tenths (0.6) per cent is fat, and not less than eight (8) per cent is nitrogen. The nitrogenous compounds contain not less than forty (40) per cent of meat bases and not less than ten (10) per cent of kreatin and kreatinin.

2. *Fluid meat extract* is identical with meat extract except that it is concentrated to a lower degree and contains not more than seventy-five (75) and not less than (50) per cent of total solids.

3. *Bone extract* is the product obtained by extracting fresh trimmed bones with boiling water and concentrating the liquid portion by evaporation after removal of fat, and contains not less than seventy-five (75) per cent of total solids.

4. *Fluid bone extract* is identical with bone extract except that it is concentrated to a lower degree and contains not more than seventy-five (75) and not less than fifty (50) per cent of total solids.

5. *Meat juice* is the fluid portion of muscle fibre, obtained by pressure or otherwise, and may be concentrated by evaporation at a temperature below the coagulating point of the soluble proteids. The solids contain not more than fifteen (15) per cent of ash not more than two and five-tenths (2.5) per cent of sodium chlorid (calculated from the total chlorin present) not more than four (4) nor less than two (2) per cent of phosphoric acid (P_2O_5), and not less than twelve (12) per cent of nitrogen. The nitrogenous bodies contain not less than thirty-five (35) per cent of coagulable proteids and not more than forty (40) per cent of meat bases.

6. *Peptones* are products prepared by the digestion of proteid material by means of enzymes or otherwise, and contain not less than ninety (90) per cent of proteoses and peptones.

7. *Gelatin (edible gelatine)* is the purified, dried, inodorous product of the hydrolysis, by treatment with boiling water, of certain tissues, as skin, ligaments, and bones, from sound animals, and contains not more than fifteen (15) per cent and not less than two (2) per cent of nitrogen.

Sauces. Must be made from sound and wholesome materials. The use of a filler is prohibited. Must contain no sweetening material other than pure sugar. Must not contain added salicylic acid, benzoic acid, saccharin, boric acid, formaldehyde, chemical preservatives or their derivatives or coloring matter. If distilled vinegar is used, it shall be so stated on the label.

Pickles. Must be made from sound and wholesome materials. Must contain no sweetening agent other than pure sugar. Must not contain added salicylic acid, benzoic acid, saccharin, boric acid, formaldehyde, chemical preservative or their derivatives, copper salts, alum, iron salts or coloring matter. If distilled vinegar is used it must be so stated on the label.

Red pepper sauce. Must be made from sound, ripe, wholesome *Red pepper*, and must contain no added filling; must not contain added salicylic acid, benzoic acid, saccharin, boric acid, formaldehyde, chemical preservatives or their derivatives or coloring matter. If distilled vinegar is used it must be so stated on the label.

Catsup. Must be made from ripe, wholesome and sound vegetable materials. The use of a filler of starch or other matter is prohibited. Must not contain added salicylic acid, benzoic acid, saccharin, boric acid, formaldehyde, or their derivatives, nor any added chemical preservative or coloring matter. If distilled vinegar is used it shall be so stated on the label.

F. *Beverages.* a. *Fruit juices—fresh, sweet, and fermented.* *Fresh fruit juices.* 1. *Fresh fruit juices* are the clean, unfermented liquid products ob-

tained by the pressing of fresh, ripe fruits, and correspond in name to the fruits from which they are obtained.

2. *Apple juice, apple must, sweet cider*, is the fresh fruit juice obtained from apples, the fruit of *Pyrus malus*, has a specific gravity (20° C.) not less than 1.0415 nor greater than 1.0690; and contains in one hundred (100) cubic centimetres (20° C.) not less than six (6) grams, and not more than twenty (20) grams of total sugars, in terms of reducing sugars, not less than twenty-four (24) centigrams nor more than sixty (60) centigrams of apple ash, which contains not less than fifty (50) per cent of potassium carbonate.

3. *Grape juice, grape must*, is the fresh fruit juice obtained from grapes (*Vitis* species), has a specific gravity (20° C.) not less than 1.0400 and not exceeding 1.1240; and contains in one hundred (100) cubic centimetres (20° C.), not less than seven (7) grams nor more than twenty-eight (28) grams of total sugars, in terms of reducing sugars, not less than twenty (20) centigrams and not more than fifty-five (55) centigrams of grape ash, and not less than fifteen (15) milligrams nor more than seventy (70) milligrams of phosphoric acid (P_2O_5).

4. *Lemon juice* is the fresh fruit juice obtained from lemon, the fruit of *Citrus limonum* Risso, has a specific gravity (20° C.) not less than 1.030 and not greater than 1.040; and contains not less than ten (10) per cent of solids, and not less than seven (7) per cent of citric acid.

5. *Pear juice, pear must, sweet perry*, is the fresh fruit juice obtained from pears, the fruit of *Pyrus communis* or *P. sinensis*.

Sterilized fruit juices. 1. *Sterilized fruit juices* are the products obtained by heating fresh fruit juices sufficiently to kill all the organisms present, and correspond in name to the fruits from which they are obtained.

Concentrated fruit juices. 1. *Concentrated fruit juices* are clean, sound fruit juices from which a considerable portion of the water has been evaporated, and correspond in name to the fruits from which they are obtained.

Sweet fruit juices, sweetened fruit juices, fruit sirups. 1. *Sweet fruit juices, sweetened fruit juices, fruit sirups*, are the products obtained by adding sugar (sucrose) to fresh fruit juices, and correspond in name to the fruit from which they are obtained.

2. *Sterilized fruit sirups* are the products obtained by the addition of sugar (sucrose) to fresh fruit juices and heating them sufficiently to kill all organisms present, and correspond in name to the fruits from which they are obtained.

9. *Cider, hard cider*, is the product made by the normal alcoholic fermentation of apple juice, and the usual cellar treatment, and contains not more than seven (7) per cent by volume of alcohol, and, in one hundred (100) cubic centimetres of the cider, not less than two (2) grams nor more than twelve (12) grams of solids, not more than eight (8) grams of sugars, in terms of reducing sugars, and not less than twenty (20) centigrams nor more than forty (40) centigrams of cider ash.

10. *Sparkling cider, champagne cider*, is cider in which the after-part of the fermentation is completed in closed containers, with or without the addition of cider or sugar liquor, and contains in one hundred (100) cubic centimetres, not less than twenty (20) centigrams of cider ash.

b. *Mead, root beer, etc. Mead.* The materials used shall be pure and wholesome according to the standards set forth in these regulations. The water used shall be potable; shall not contain added salicylic acid, benzoic acid, saccharin, boric acid, formaldehyde, or their derivatives, nor any added chemical preservative or coloring matter.

Root beer shall be manufactured from roots, bark, leaves, berries, herbs, or the oils extracted therefrom, caramel, or other harmless ingredients; shall not contain added salicylic acid, saccharin, boric acid, formaldehyde, or their derivatives, or added chemical preservatives or coloring matter.

c. Malt liquors. 1. *Malt liquor* is a beverage made by the alcoholic fermentation of an infusion in potable water, of barley-malt and hops, with or without malted cereals.

2. *Beer* is a malt liquor produced by bottom fermentation, and contains in one hundred (100) cubic centimetres, at (20° C.) not less than five (5) grams of extractive matter, and sixteen one-hundredths (.16) gram of ash, chiefly potassium phosphate, and not less than two and twenty-five one hundredths (2.25) grams of alcohol.

3. *Lager beer, stored beer*, is beer which has been stored in casks for a period of at least three months, and contains in one hundred (100) cubic centimetres (at 20° C.) not less than five (5) grams of extractive matter, and sixteen one-hundredths (.16) gram of ash, chiefly potassium phosphate, and not less than two and fifty one-hundredths (2.50) grams of alcohol.

4. *Malt beer* is beer made of an infusion in potable water, of barley malt, and hops, and containing in one hundred (100) cubic centimetres (at 20° C.) not less than five (5) grams of extractive matter, nor less than two-tenths (.2) gram of ash, chiefly potassium phosphate, nor less than two and twenty-five one-hundredths (2.25) grams of alcohol, nor less than four-tenths (.4) gram of crude protein (nitrogen $\times$ 6.25).

5. *Ale* is a malt liquor produced by top fermentation and contains in one hundred (100) cubic centimetres (at 20° C.) not less than two and seventy-five one hundredths (2.75) grams of alcohol, nor less than five (5) grams of extract.

6. *Porter and stout* are varieties of ale colored by the addition of highly roasted malt to the infusion.

d. Spirituous liquors. 1. *Distilled spirits* is the distillate obtained from a fermented mash of cereals, molasses, sugars, fruits, or other starch- or sugar bearing substances, and contains all the condensed products of the fermentation volatile at the usual temperature of distillation.

2. *Rectified spirits* is distilled spirit which at the time of, or subsequent to distillation is subjected to a rectifying process by means of which a part of the volatile products of the distillation is separated from the ethyl alcohol therein.

3. *Alcohol, cologne spirit, neutral spirit, velvet spirit, or silent spirit* is distilled spirit from which all, or nearly all, its constituents are separated, except ethyl alcohol and water, and contains not less than ninety-four and nine-tenths (94.9) per cent (189.8 proof) by volume of ethyl alcohol.

4. *New whiskey* is the distilled spirits from the properly fermented mash of malt cereals, or cereals the starch of which has been hydrolized by malt, is of an alcoholic strength corresponding to the excise laws of the various countries in which it is made, and contains not less than one hundred and twenty-five (125) nor more than three hundred and fifty (350) grams of the secondary products of distillation congeneric with ethyl alcohol, not less than ninety (90) nor more than two hundred and twenty-five (225) grams of fusel oil (higher alcohols as amyllic), not more than twenty (20) grams of aldehydes, not less than fifteen (15) nor more than one hundred (100) grams of ethers (as acetic ether), not less than two (2) nor more than twenty-five (25) grams of volatile acids (as acetic) to one hundred (100) litres of proof ethyl alcohol (50 per cent ethyl alcohol by volume).

5. *Whiskey (potable whiskey)* is new whiskey which has been stored in wood for not less than four (4) years and mixed only with pure water at the time of its preparation for consumption, and contains unless otherwise prescribed by law, not less than forty-five (45) per cent of ethyl alcohol by volume, and the relative quantities of secondary products to ethyl alcohol corresponding to the varieties of whiskey under six (6) to fifteen (15), inclusive.

6. *Rye whiskey* is whiskey in the manufacture of which rye is the principal cereal used, and contains not less than two hundred (200) nor more than five hundred (500) grams of the secondary products of distillation congeneric with ethyl alcohol, not less than one hundred (100) nor more than two hundred and fifty (250) grams of fusel oil (higher alcohols as amyllic), not more than twenty-five grams of aldehydes, not less than forty (40) nor more than one hundred and fifty (150) grams of ethers (as acetic ether), not less than thirty (30) nor more than eighty-five (85) grams of volatile acids (as acetic) to one hundred (100) litres of proof ethyl alcohol (50 per cent ethyl alcohol by volume).

7. *Bourbon whiskey* is whiskey in which Indian corn (maize) is the principal cereal used, and contains not less than two hundred (200) nor more than five hundred (500) grams of the secondary products of distillation congeneric with ethyl alcohol, not less than one hundred (100) nor more than two hundred and fifty (250) grams of fusel oil (higher alcohols and amyllic), not more than twenty-five (25) grams of aldehydes, not less than forty (40) nor more than one hundred and fifty (150) grams of ethers (as acetic ether), not less than thirty (30) nor more than eighty-five (85) grams of volatile acids (as acetic) to one hundred litres of proof ethyl alcohol (50 per cent ethyl alcohol by volume).

8. *Corn whiskey* is whiskey made from maize (Indian corn), the starch of which has been hydrolized by malting or by the action of barley malt, and contains the proportions of the various ingredients specified for bourbon whiskey.

9. *Arrack* is distilled spirit made from rice.

10. *Blended whiskey* is a mixture of two or more whiskeys, and contains the relative quantities of secondary products to ethyl alcohol of the varieties of whiskey forming the blend.

11. *Rectified new whiskey* is new whiskey deprived of a part of its secondary volatile products, and contains not less than sixty (60) grams of the secondary volatile products of distillation congeneric with ethyl alcohol, not less than forty (40) grams of fusel oil (higher alcohol as amyllic) not more than eight (8) grams of aldehydes, not less than five (5) grams of ethers (as acetic ether), not less than one (1) gram of volatile acids (as acetic) to one hundred (100) litres of proof ethyl alcohol (50 per cent of ethyl alcohol by volume).

12. *Rectified whiskey* is rectified new whiskey stored in wood not less than three (3) years, except where otherwise prescribed by law, and contains not less than one hundred (100) grams of the secondary products of distillation congeneric with ethyl alcohol, not less than fifty (50) grams of fusel oil (higher alcohols as amyllic), not more than ten (10) grams of aldehydes, not less than twenty (20) grams of ethers (as acetic ether), not less than fifteen (15) grams of volatile acids (as acetic) to one hundred (100) litres of proof ethyl alcohol (50 per cent ethyl alcohol by volume).

13. *Scotch new whiskey* is whiskey made in Scotland solely from barley malt in the drying of which over burning peat a smoky or peaty flavor is imparted to the product, and contains not less than one hundred and twenty-five (125) nor more than three hundred and fifty (350) grams of the secondary product of distillation congeneric with ethyl alcohol, not less than ninety (90) nor more

than two hundred and twenty-five (225) grams of fusel oil (higher alcohols as amyllic), not more than twenty (20) grams of aldehydes, not less than fifteen (15) nor more than one hundred (100) grams of ethers (as acetic ether), not less than two (2) nor more than twenty-five (25) grams of volatile acids (as acetic) to one hundred (100) litres of proof ethyl alcohol (50 per cent ethyl alcohol by volume).

14. *Scotch whiskey* is Scotch new whiskey which has been stored in wood for not less than four years and mixed only with pure water at the time of its preparation for consumption, and contains not less than one hundred and fifty (150) nor more than four hundred and fifty (450) grams of the secondary products of distillation congeneric with ethyl alcohol, not less than one hundred (100) nor more than two hundred and fifty (250) grams of fusel oil (higher alcohols as amyllic) not more than twenty-five (25) grams of aldehyde, not less than twenty-five (25) nor more than one hundred and twenty-five (125) grams of ethers (as acetic ether), not less than ten (10) nor more than forty (40) grams of volatile acids (as acetic) to one hundred (100) litres of proof ethyl alcohol (50 per cent ethyl alcohol by volume).

15. *Irish new whiskey* is whiskey made in Ireland either from barley malt, or malt and unmalted barley, or other cereals, and contains not less than one hundred and twenty-five (125) nor more than three hundred and fifty (350) grams of the secondary products of distillation congeneric with ethyl alcohol, not less than ninety (90) nor more than two hundred and twenty-five (225) grams of fusel oil (higher alcohols as amyllic), not more than twenty (20) grams of aldehydes, not less than fifteen nor more than one hundred (100) grams of ethers (as acetic ether), not less than two (2) nor more than twenty-five (25) grams of volatile acids (as acetic) to one hundred (100) litres of proof ethyl alcohol (50 per cent ethyl alcohol by volume).

16. *Irish whiskey* is Irish new whiskey which has been stored in wood for not less than four years and mixed only with pure water at the time of its preparation for consumption, and contains not less than one hundred and fifty (150) nor more than four hundred and fifty (450) grams of the secondary products of distillation congeneric with ethyl alcohol not less than one hundred (100) nor more than two hundred and fifty (250) grams of fusel oil (higher alcohols as amyllic), not more than twenty-five grams of aldehydes, not less than twenty-five (25) nor more than one hundred and twenty-five (125) grams of ethers (as acetic ether), not less than ten (10) nor more than forty (40) grams of volatile acids (as acetic) to one hundred (100) litres of proof ethyl alcohol (50 per cent ethyl alcohol by volume.)

17. *New rum* is distilled spirits made from the fermented juice of the sugar cane, the massecuite made therefrom, molasses from the massecuite or any intermediate product save sugar, and contains not less than one hundred and twenty-five (125) nor more than three hundred and fifty (350) grams of the secondary products of distillation congeneric with ethyl alcohol, not less than sixty (60) nor more than one hundred and fifty (150) grams of fusel oil (higher alcohols as amyllic) not more than thirty (30) grams of aldehydes, not less than thirty (30) nor more than one hundred (100) grams of ethers (as acetic ether), not less than twenty (20) nor more than (50) grams of volatile acids (as acetic) to one hundred (100) litres of proof ethyl alcohol (50 per cent ethyl alcohol by volume).

18. *Rum* is new rum stored not less than four (4) years in wood, and contains not less than one hundred and seventy-five (175) nor more than five hundred (500) grams of the secondary products of distillation congeneric with ethyl alcohol, not less than eighty (80) nor more than two hundred (200) grams of

fusel oil (higher alcohols as amyllic), not more than forty (40) grams of aldehydes, not less than fifty (50) nor more than one hundred and fifty (150) grams of ethers (as acetic ether) not less than thirty-five (35) nor more than one hundred (100) grams of volatile acids (as acetic) to one hundred (100) litres of proof ethyl alcohol (50 per cent ethyl alcohol by volume).

19. *New brandy* is a distilled spirit made from sound potable wine, and contains not less than one hundred and twenty-five (125) nor more than three hundred and fifty (350) grams of the secondary products of distillation congeneric with ethyl alcohol, not less than seventy (70) nor more than one hundred and fifty (150) grams of fusel oil (higher alcohols as amyllic), nor more than twenty (20) grams of aldehydes, not less than thirty (30) nor more than one hundred (100) grams of ethers (as acetic ether), not less than five (5) nor more than twenty (20) grams of volatile acids (as acetic) to one hundred (100) litres of proof ethyl alcohol (50 per cent ethyl alcohol by volume).

20. *Brandy* is new brandy stored in wood for not less than four (4) years, and contains not less than one hundred and fifty (150) nor more than five hundred (500) grams of the secondary products of distillation congeneric with ethyl alcohol, not less than eighty (80) nor more than two hundred (200) grams of fusel oil (higher alcohols as amyllic), not more than thirty (30) grams of aldehydes, not less than thirty-five (35) nor more than one hundred and fifty (150) grams of ethers (as acetic ether), not less than thirty (30) nor more than one hundred (100) grams of volatile acids (as acetic) to one hundred (100) litres of proof ethyl alcohol (50 per cent ethyl alcohol by volume).

21. *Cognac* is brandy prepared in the departments of the Charente, France, from pure, sound wine produced in those departments.

e. Carbonated waters. Water—Potable—Water to be potable must be suitable to all forms of domestic use; must possess no objectionable smell or taste; must be free from animal, especially human refuse material; must be free from vegetable material in a state of active decomposition; must be free from pathogenic bacteria; must be free from such an amount of suspended material of whatever character as would make it unsightly in appearance and unsuited to the ordinary industrial uses of a community.

Carbonated waters are waters charged with carbonic acid gas, and may be naturally carbonated or artificially carbonated. Label must state how carbonated, and if the source of the water is given thereon, the water must be true to its label. All carbonated waters must be wholesome and potable.

Spring and well waters are waters derived from springs or wells; they must be potable and wholesome; they may or may not be medicinal; and must come from the well or spring indicated on the label and no other. The standard for any spring or well water will be the water itself, sample being taken at its source by a representative of this Board.

Artificial mineral waters must be so labeled, and the water used in their manufacture must be wholesome and potable. All waters must be true to label, and if an analysis is published as an advertisement, or is placed on the label, the water must conform thereto.

G. Vinegar. * * *

6. *Spirit vinegar, distilled vinegar, grain vinegar*, is the product made by the acetous fermentations of dilute distilled alcohol, and contains, in one hundred (100) cubic centimetres (20° C.), not less than four (4) grams of acetic acid, and shall be free from coloring matter, added during or after distillation, and from color other than that imparted to it by distillation.

Bread and yeast. Bread must be made of pure and wholesome materials as provided for in these regulations, must not contain adulterants, alums, or cop-

per salts, should not contain more than forty (40) per cent of water nor have an acidity in ten (10) grams of fresh bread requiring more than 10 c. c. of 1-10 normal sodium hydroxide solution to neutralize it.

Compressed Yeast should be used when fresh. Such yeast should have a creamy white color, uniform throughout, should possess a fine even texture, should be moist without being slimy, should not have a "cheesy" odor, such odor indicating decomposition as does a dark streaked color.

IV. PRESERVATIVES AND COLORING MATTERS.

Standard preservatives are salt, sugar, vinegar, spices, and their essential oils, wood smoke, edible oils and fats, and alcohol.

The use in food products, of any other preservatives or antiseptics, or of any substance which preserves or enhances the natural color of a food product, or of a coloring matter is prohibited except as provided for in these Regulations.

* * *

See Reg. 14, Section (u) [and F. I. D. 76 as to permitted coal tar dyes.]

REG. 46. *Taking orders deemed a sale.* Taking orders for same. The taking of orders or the making of agreements or contracts by any person, firm or corporation, or by any agent or representative thereof, for the future delivery of any of the articles, products, goods, wares, or merchandise embraced within the provisions of these Regulations, shall be deemed a sale within the meaning of these Regulations.

REG. 47. *Person defined.* The word "person," as used in these regulations shall be construed to import both the plural and the singular, as the case demands, and shall include corporations, companies, societies, and associations, when construing and enforcing the provisions of these regulations the act, omission or failure of any officer, agent or other person acting for or employed by any corporation within the scope of his employment or office, shall in every case be also deemed to be the act, omission, or failure of such corporation, company, society, or association, as well as that of the person.

REG. 48. *Penalty.* Any person convicted of violating any of the provisions of the foregoing Regulations wherein penalty is not provided, shall be punished pursuant to the provisions of Section 3, Act 98 of 1906.

These Regulations shall be in force and effect from and after their adoption and promulgation by the State Board of Health.

The State Board of Health reserves the right conferred on it by Section 2, Act 98 of 1906, "to further revise and amend" whenever the interests of the public health, the advancement of scientific knowledge, or the rulings of the National Food Department make it advisable so to do.

All laws and regulations in conflict with these Regulations are hereby repealed.

Adopted April 25, 1908.

MARYLAND.

FRUITS, ETC.

Sec. 1. *Fruits and vegetables to be marked.* All shippers and sellers of all fruits and vegetables in Wicomico county shall be compelled to stamp or mark all baskets, barrels, boxes, packages, crates, parcels or other receptacles used by them for the shipment or sale of any fruit, fruits or vegetables with his, her or their name or names, initials, or with some distinguishing device or mark which may be readily and easily read and seen on the same before such fruit, fruits or vegetables shall be offered for shipment or sale; and if any shipper or seller of any fruit, fruits or vegetables, shall neglect or fail to comply with the provisions of this section, he or she, or they, shall pay a fine of five dollars; said fine to be applied to the public school fund for Wicomico county, but nothing in this Act shall apply to hucksters selling in quantities less than full packages, or to anything delivered to canneries.

Sec. 2. *Effect.* This Act shall take effect from May 1, 1908.

Approved April 6, 1908. Laws of 1908, art. 23, ch. 712, p. 1125.

MASSACHUSETTS.

GENERAL FOOD LAWS.^a

SEC. 70. General inspection authority. Boards of health of cities and towns, by themselves, their officers or agents, may inspect the carcasses of all slaughtered animals and all meat, fish, vegetables, produce, fruit or provisions of any kind found in their cities or towns, and for such purpose may enter any building, enclosure or other place in which such carcasses or articles are stored, kept or exposed for sale. If, on such inspection, it is found that such carcasses or articles are tainted, diseased, corrupted, decayed, unwholesome or, from any cause, unfit for food, the board of health shall seize the same and cause it or them to be destroyed forthwith or disposed of otherwise than for food. All money received by the board of health for property disposed of as aforesaid shall, after deducting the expenses of said seizure, be paid to the owner of such property. If the board of health seizes or condemns any such carcass or meat for the reason that it is infected with a contagious disease, it shall immediately give notice to the board of cattle commissioners of the name of the owner or person in whose possession it was found, the nature of the disease and the disposition made of said meat or carcass.—*As amended April 17, 1908, Acts and Resolves of 1908, ch. 411, p. 276. See Bul. 69, Rev., pt. 3, p. 266.*

SEC. 72. Penalty for hindering inspectors. Whoever prevents, obstructs or interferes with the board of health, its officers or agents, in the performance of its duties as provided herein, or hinders, obstructs or interferes with any inspection or examination by it or them, or whoever secretes or removes any carcass, meat, fish, vegetables, fruit or provisions of any kind, for the purpose of preventing the same from being inspected or examined under the provisions of sections seventy to seventy-six, inclusive, shall be punished by a fine of not more than one hundred dollars or by imprisonment for not more than sixty days, or by both such fine and imprisonment.—*As amended April 17, 1908; Acts and Resolves of 1908, ch. 411, p. 276. See Bul. 69, Rev., pt. 3, p. 266.*

Revised Laws, 1902, vol. 1, ch. 56, p. 555.

SEC. 1. Repeal. Sections twenty-five and twenty-six of chapter seventy-five of the Revised Laws (Bul. 69, Rev., pt. 3, p. 248), relating to the sale of adulterated food and drugs, are hereby repealed.

SEC. 2. Effect. This act shall take effect upon its passage.

Approved March 18, 1908. Acts and Resolves of 1908, ch. 238, p. 153.

BREAD.

SEC. 6. Penalty. Whoever violates any provision of the preceding three sections shall be punished by a fine of not more than ten dollars for each offence. The sealer of weights and measures in the respective cities and towns, or the commissioner of weights and measures of the commonwealth, shall cause the provisions of the said three sections to be enforced.—*As amended March 10, 1908; Acts and Resolves of 1908, ch. 197, p. 114. See Bul. 69, Rev., pt. 3, p. 252.*

Revised Laws 1902, vol. 1, ch. 57, pp. 557-8.

^a See also Meat, page 39.

MEAT.^a

SEC. 1. *Prohibition; penalty.* The sale, offer or exposure for sale, or delivery for use as food, of the carcass, or any part or product thereof, of any animal which has come to its death in any manner or by any means otherwise than by slaughter or killing while in a healthy condition, or which at the time of its death is unfit by reason of disease, exhaustion, abuse, neglect or otherwise for use as food, or of any calf weighing less than forty pounds when dressed, with head, feet, hide and entrails removed, is hereby declared to be unlawful and prohibited. Whoever sells or offers or exposes for sale or delivers or causes or authorizes to be sold, offered or exposed for sale or delivered for use as food any such carcass or any part or product thereof, shall be punished by fine of not more than two hundred dollars or by imprisonment for not more than six months.

SEC. 2. *Inspectors of state and municipal boards of health; seizure and destruction of unlawful products.* The state board of health and its inspectors, and the state inspectors of health and all boards of health of cities and towns and their inspectors, officers, agents and assistants in their respective districts, shall have and exercise the same powers and duties in and for the enforcement of this act as are at any time conferred or imposed by law upon any board of health, inspector, officer, agent or assistant in respect of any other article or substance the sale or use of which for food is unlawful or prohibited; and it shall be their duty to seize any such carcass or part or product thereof as described in section one hereof, and cause the same to be destroyed forthwith or disposed of otherwise than for food; and all moneys received by any board of health for any property so disposed of shall, after deducting the expenses of such seizure and disposal, be paid to the owner of such property if known.

SEC. 3. *Places to be inspected.* Such inspectors, officers, agents and assistants shall visit and keep under observation all places within their respective districts at which neat cattle, sheep, swine or other animals intended for slaughter or for sale or use as food are delivered from transportation, and shall have at all times free access to all such places and to all railroad trains or cars or other vehicles in which such animals may be transported, for the purpose of preventing violations of this act and of detecting and punishing the same.

SEC. 4. *Powers of inspection.* The state inspectors of health in their respective districts, and the inspectors appointed by the state board of health for duties relative to the sale of food and drugs, shall have the same rights, powers and authority for and in respect of the inspection, seizure and disposition of all carcasses, meats and provisions which are tainted, diseased, corrupted, decayed, unwholesome, or from any cause unfit for food, or the sale of which for food is unlawful, as are conferred by sections seventy and seventy-one of chapter fifty-six and by section one hundred and two of chapter seventy-five of the Revised Laws, or by other laws, upon boards of health of cities and towns or their inspectors in respect of the articles therein specified; with power to prosecute all offences relating thereto.

SEC. 5. *Inspection of slaughter houses.* In addition to the supervision now provided for by law, all slaughter houses shall be under the supervision of the state board of health and subject to inspection by the state inspectors of health in their respective districts.

SEC. 6. *Amendment.* Section one hundred and five of chapter seventy-five of the Revised Laws, as amended by section two of chapter three hundred and twelve of the acts of the year nineteen hundred and two, and by section two of

^a See also General Food Law, page 38.

chapter two hundred and twenty of the acts of the year nineteen hundred and three, is hereby further amended by striking out all after the word "old," in the seventh line, so as to read as follows: Sec. 105. *Exemption.* The provisions of the six preceding sections shall not apply to a person not engaged in such business, who, upon his own premises and not in a slaughter house, slaughters his own neat cattle, sheep or swine, but the carcass of any such animals shall be inspected by an inspector at the time of slaughter, unless said animal is less than six months old.

SEC. 7. *Authority of other officers unimpaired.* Nothing in this act shall affect or impair the rights, powers or authority of any board or officer not herein mentioned.

Approved March 31, 1908. Acts and Resolves of 1908, ch. 329, pp. 218-20.

SEC. 71. *Inspection of veal.* The board of health, by themselves, their officers or agents, may inspect all veal found, offered or exposed for sale or kept with the intent to sell in its city or town, and if, in its opinion, said veal is that of a calf less than four weeks old when killed, the board shall seize and destroy or dispose of it as provided in the preceding section, subject, however, to the provisions thereof relative to the disposal of money.—*As amended April 17, 1908; Acts and Resolves of 1908, ch. 411, p. 276. See Bul. 69, Rev., pt. 3, p. 266.*

Revised Laws, 1902, vol. 1, ch. 56, p. 555.

MILK.

SEC. 3. *Unclean vessels without name of owner; penalty.* Every licensed milk dealer who sells, or has in his possession with intent to sell, milk not contained in clean vessels bearing his own name, or the name under which his business is conducted, and bearing no other name, shall be punished by a fine of ten dollars for each offence; but the provisions of this section shall not apply to persons using clean vessels bearing the name of another person whose written permission for such use shall have been obtained previously and registered in the office of the milk inspector, in municipalities having such officer, and in other municipalities registered in the office of the city or town clerk.—*As amended April 22, 1908; Acts and Resolves of 1908, ch. 435, p. 292. See Bul. 104, p. 33.*

Approved March 1, 1906. Acts and Resolves 1906, ch. 116, p. 62.

SEC. 2. *Repeal.* Section four (Bul. 104, p. 33) of said chapter one hundred and sixteen is hereby repealed.

Approved April 22, 1908. Acts and Resolves of 1908, ch. 435, p. 292.

- SEC. 12. *Expenditures; report.* The bureau may expend not more than eight thousand dollars annually in its work, and it may co-operate with the state board of health and with inspectors of milk, but it shall not interfere with the duties of such board or officers. It shall annually, before the fifteenth day of January, report to the general court in detail the number of agents, assistants, experts and chemists employed by it, with their expenses and disbursements, of all investigations made by it, of all cases prosecuted with the results thereof, and other information advantageous to the dairy industry.—*As amended April 17, 1908; Acts and Resolves of 1908, ch. 416, p. 278. See Bul. 69, Rev., pt. 3, p. 253.*

Revised Laws 1902, vol. 1, ch. 89, pp. 778-9.

SEC. 56. *Standard for milk.* In prosecutions under the provisions of sections fifty-one to sixty-four, inclusive, milk which, upon analysis, is shown to contain less than twelve and fifteen hundredths per cent of milk solids or less than three and thirty-five hundredths per cent of fat, shall not be considered of good standard quality.—*As amended June 13, 1908; Acts and Resolves of 1908, ch. 643, p. 566. See Bul. 69, Rev., pt. 3, p. 258.*

Revised Laws 1902, vol. 1, ch. 56, pp. 547-54.

SEC. 1. *Heated milk; fine if not labeled.* Whoever, himself or by his servant or agent, or as the servant or agent of any person, firm or corporation, sells, exchanges or delivers or has in his custody or possession with intent to sell, exchange or deliver any milk which has been subjected to artificial heat greater than one hundred and sixty-seven degrees Fahrenheit, not having the words "heated milk" distinctly marked upon a light ground in plain black uncondensed gothic letters at least one inch in length in a conspicuous place upon every vessel, can or package from or in which such milk is, or is intended to be, sold, exchanged or delivered shall for a first offence be punished by a fine of not less than fifty nor more than two hundred dollars, for a second offence by a fine of not less than one hundred nor more than three hundred dollars, and for a subsequent offence by a fine of fifty dollars and by imprisonment for not less than sixty nor more than ninety days. If such vessel, can or package is of the capacity of not more than two quarts, said words may be placed upon a detachable label or tag attached thereto and said letters may be less than one inch in length, but not smaller than brevier gothic capital letters.

SEC. 2. *Exemptions.* Nothing in this act shall be construed as applying to condensed milk or to milk which has been concentrated to one-half its volume or less.

Approved June 1, 1908. Acts and Resolves of Massachusetts, 1908, ch. 570, p. 404.

WATER.

SEC. 1. *Defiling water supply.* Any police officer or constable of a city or town in which any pond, stream or reservoir used for the purpose of domestic water supply is wholly or partly situated, acting within the limits of his city or town, and any executive officer of a water board, board of water commissioners, public institution or water company, furnishing water for domestic purposes, or agent of such water board, board of water commissioners, public institution or water company, duly authorized in writing therefor by such boards, institution or company, acting upon the premises of such board, institution or company and not more than five rods from the water, for such supply may, without a warrant, arrest any person found in the act of bathing in a pond, stream or reservoir, the water of which is used for the purpose aforesaid, and detain him in some convenient place until a complaint can be made against him therefor.

SEC. 2. *Effect.* This act shall take effect upon its passage.

Approved May 28, 1908. Acts and Resolves of 1908, ch. 539, pp. 377-78.

MISSISSIPPI.

SEC. 2. *Repeal.* That sections * * * 1766 * * * of the Mississippi Code of 1906 be, and the same are hereby, repealed.—*Repealed February 19, 1908; Laws of 1908, ch. 115, p. 118. See Bul. 69, Rev., pt. 4, p. 326.*

Annotated Code, 1892, ch. 37, p. 430, or Code of 1906, sec. 1766.

NEW JERSEY.

GENERAL FOOD LAWS.

SEC. 3. Adulteration defined. For the purposes of this act an article shall be deemed to be adulterated * * *

In the case of confectionery:

It it contains terra alba, barytes, talc, chrome yellow or other mineral substance, or poisonous color or flavor, or other ingredient deleterious or detrimental to health, or any vinous, malt or spirituous liquor or compound or narcotic drug.

In the case of food:

First. If any substance has been mixed or packed with it so as to reduce or lower or injuriously affect its quality or strength.

Second. If any substance has been substituted wholly or in part for the article.

Third. If any valuable constituent of the article has been wholly or in part abstracted.

Fourth. If it be mixed, colored, powdered, coated or stained in a manner whereby damage or inferiority is concealed.

Fifth. If it contain any added poisonous or other added deleterious ingredient which may render such article injurious to health; *provided*, that when in the preparation of food products for shipment they are preserved by any external application applied in such manner that the preservative is necessarily removed mechanically, or by maceration in water, or otherwise, and directions for the removal of said preservative shall be printed on the covering or the package, the provisions of this act shall be construed as applying only when said products are ready for consumption.

Sixth. If it consists in whole or in part of a filthy, decomposed or putrid animal or vegetable substance, or any portion of an animal unfit for food, whether manufactured or not, or if it is the product of a diseased animal, or one that has died otherwise than by slaughter.—*As amended April 16, 1908, Acts of 1908, ch. 308, pp. 629-630. See Bul. 112, pt. 2, pp. 7-8.*

SEC. 4. Misbranding defined. The term "misbranded," as used herein, shall apply to all drugs, or articles of food, or articles which enter into the composition of food, the package or label of which shall bear any statement, design or device regarding such article, or the ingredients or substances contained therein, which shall be false or misleading in any particular, and to any food or drug product which is falsely branded as to the State, Territory or country in which it is manufactured or produced.

For the purposes of this act an article shall also be deemed to be misbranded * * *

In the case of food:

First. If it be an imitation of or offered for sale under the distinctive name of another article.

Second. If it be labeled or branded so as to deceive or mislead the purchaser, or purport to be a foreign product when not so, or if the contents of the package as originally put up shall have been removed, in whole or in part, and other con-

tents shall have been placed in such package, or if it fail to bear a statement on the label of the quantity or proportion of any morphine, opium, cocaine, heroin, alpha or beta eucaine, chloroform, cannabis indica, chloral,^a hydrate, acetanilide, acetphenetidine, or phenacetin or antipyrin, or any derivative or preparation of any such substances contained therein.

Third. If in package form, and the contents are stated in terms of weight or measure, they are not plainly and correctly stated on the outside of the package.

Fourth. If the package containing it, or its label shall bear any statement, design or device regarding the ingredients or substances contained therein, which statement, design or device shall be false or misleading in any particular.—*As amended April 16, 1908; Acts of 1908, ch. 308, pp. 630-632. See Bul. 112, pt. 2, p. 8.*

SEC. 46. Guarantee for protection of dealer. No dealer shall be prosecuted under the provisions of this act for distributing or selling, or having in his possession with intent to distribute or sell, any article of food or drug which under any of said provisions shall be deemed to be adulterated or misbranded; *provided*, that said article of food or drug is distributed or sold or had in possession with intent to distribute or sell in the original unbroken package in which it was received by said dealer, and that, in case said article was purchased by said dealer from a wholesaler, jobber, manufacturer, or other person residing in this State, and said dealer can establish a guarantee signed by such wholesaler, jobber, manufacturer or other person from whom he purchased such article, to the effect that the same is not adulterated or misbranded within the meaning of this act, designating it; or in case said article was purchased by said dealer from a wholesaler, jobber, manufacturer or other person residing in the United States of America, but outside of this State, and said dealer can establish a guarantee, signed by such wholesaler, jobber, manufacturer or other person from whom he purchased such article, to the effect that the same is not adulterated or misbranded within the meaning of an act of the Congress of the United States of America, entitled "An act for preventing the manufacture, sale or transportation of adulterated or misbranded, or poisonous or deleterious foods, drugs, medicines and liquors, and for regulating traffic therein, and for other purposes," approved June thirtieth, one thousand nine hundred and six, and the supplements and amendments thereof. Such guaranty, to afford protection, shall contain the name and address of the person making the sale of such article to such dealer, and in such case said person, if he is a resident of this State, shall be amenable to the prosecution, fines and other penalties which would attach in due course to the dealer under the provisions of this act. If the guaranty is signed by a person who resides outside of this State, then the Board of Health of this State shall report the facts in the case to the Secretary of Agriculture of the United States, or the proper officer appointed for the enforcement of the above-mentioned act of Congress; *and provided further*, that no guarantee that any article is not adulterated or misbranded within the meaning of the above-mentioned act of Congress, shall be effective to exempt any dealer from prosecution under this act, unless the provisions of the above-mentioned act of Congress and of this act covering the adulteration and misbranding of such guaranteed article are identical.

The provisions of this act relating to misbranding shall not apply to the distribution or sale or to the possession with intent to distribute or sell by any dealer of such proprietary foods and medicines as were in such dealer's stock in this State on October first, nineteen hundred and eight; *provided*, that the package or other container in which such foods or medicines shall be contained

^a So in Statutes.

shall be plainly and conspicuously marked with the words and figures "On hand Oct. 1st, 1908."—*As amended April 16, 1908; Acts of 1908, ch. 308, pp. 632-633. See Bul. 112, pt. 2, pp. 12-13.*

Approved May 20, 1907. Acts of 1907, ch. 217, pp. 485-502.

SEC. 5. Exemptions for exports; preservatives. No article shall be deemed to be adulterated or misbranded within the meaning of this act when specially prepared for export to any foreign country, if such article shall be prepared and packed according to the directions of the foreign purchaser, and if no substance is used in the preparation or packing of such article which is prohibited by the laws of the foreign country for export to which said article was prepared; *provided*, that if such article shall be sold or offered for sale for use or consumption within the United States of America, then all the provisions of this act, with regard to adulteration and misbranding, shall apply thereto; *and provided further*, that all food products manufactured in this State during the years one thousand nine hundred and seven and one thousand nine hundred and eight, in which preservatives are used, which preservatives are not now specifically prohibited by the Department of Agriculture of the United States, shall be exempt from the provisions of this act; *provided*, the use of such preservatives is stated upon the label or in branding such products, and also the date of their manufacture.—*As amended April 13, 1908; Acts of 1908, ch. 242, pp. 447-478. See Bul. 112, pt. 2, sec. 6 (5), p. 8.*

Approved May 20, 1907. Laws of 1907, ch. 217, p. 488.

CONFECTIONERY.

See General Food Laws, page 43.

MILK.

SEC. 6. Standard; modified milk. No person shall distribute or sell, or have in his possession with intent to distribute or sell, any milk which contains less than twelve per centum of milk solids, or more than eighty-eight per cent. of watery fluids, or less than three per centum of milk fats; *provided, however*, that it shall not be unlawful for any person to distribute or sell, or have in his possession with intent to distribute or sell, in a container having a capacity of not more than twelve fluid ounces, milk especially prepared for infant or invalid feeding by adding thereto pure water, lime water, milk sugar, cereal starches, or other substances which shall not differ in purity, quality or strength from the standard fixed by this act, or by removing therefrom the sugar or any part thereof, if every such container have blown or moulded in it the words "modified milk" in letters which shall not be less than one-quarter inch in height and the several lines of which shall not be less than one-sixteenth of an inch in width; *and, provided also*, that the milk in such container, before modification, shall have been milk of the standard fixed by this act.—*As amended April 14, 1908; Laws of 1908, ch. 260, p. 551. See Bul. 112, pt. 2, p. 15.*

SEC. 8. Adulterated or unclean milk prohibited. No person shall distribute or sell or have in his possession with intent to distribute or sell any milk or cream which contains any water, drug, chemical, preservative, coloring matter, condensed milk, or any substance of any kind or character which has been added thereto or mixed therewith; *provided, however*, it shall not be unlawful for any person to distribute or sell or have in his possession with intent to distribute or sell, any milk or cream modified especially for infant or invalid feeding, by adding thereto or mixing therewith pure water, lime water, milk sugar, cereal starches

or other substances, as provided for in section six of this act, if such modified milk shall be in a container having a capacity of not more than twelve fluid ounces, which container shall be marked as provided for in section six of this act. No person shall distribute or sell, or have in his possession with intent to distribute or sell any milk or cream which is the product in whole or in any part of any animal kept in a crowded, uncleanly or unhealthy place or condition, or which is the product in whole or in part of any animal fed on swill, or any substance in a state of rottenness or putrefaction, or on any substance of an unwholesome nature, or on any food or substance which may produce diseased or unwholesome milk. No person shall distribute or sell, or have in his possession with intent to distribute or sell, any milk or cream which is produced in whole or in part from any animal within fifteen days before or five days after parturition.—*As amended April 14, 1908; Laws of 1908, ch. 260, p. 552. See Bul. 112, pt. 2, p. 15.*

Approved May 20, 1907. Acts of 1907, ch. 217, pp. 488-499.

NEW YORK.

DAIRY PRODUCTS.

See Appendix, Bulletin 112, Part II, page 148, for law regulating dairy products, approved July 18, 1907, and included in the compilation for the year ending June 30, 1907, for convenience.

FRUIT.

SEC. 185. *Other than standard apples prohibited.* No person shall buy for resale, sell, or expose or offer for sale as and for evaporated apples any evaporated apples intended to be used for food, or for consumption by any person other than standard evaporated apples.—*As amended May 23, 1908; Laws of 1908, vol. 2, ch. 486, p. 1704. See Bul. 112, pt. 2, p. 21.*

SEC. 186. *Standard evaporated apples defined.* Evaporated apples containing not more than twenty-seven per centum of water or fluids as determined by drying for four hours at the temperature of boiling water shall be considered standard evaporated apples for the purposes of this act.—*As amended May 23, 1908; Laws of 1908, vol. 2, ch. 486, p. 1704. See Bul. 69, Rev., pt. 5, p. 428.*

SEC. 187. *Only New York fruit to be so labeled.* No person or persons shall sell, offer or expose for sale apples, pears or peaches as and for New York state grown apples, pears or peaches if they were not grown or produced within the state of New York; nor shall they brand or label the package or barrel containing such apples, pears or peaches as New York state apples, pears or peaches if they were not grown or produced within the state of New York. Any person or persons packing or repacking or causing apples or pears to be packed or repacked to be sold upon the markets, shall pack or repack or cause them to be packed or repacked in such a manner that each separate package or barrel shall be packed substantially uniform without intent to deceive the purchaser. Any person, persons or corporation buying from a grower apples or pears which are packed in packages or barrels, marked or labeled with the name of the grower who causes such apples or pears to be repacked in the same packages or barrels or who uses the same packages or barrels for the packing of other fruit or apples or pears shall erase from such package or barrel the name of the grower or packer first or originally placed thereon. But the facing of such package or barrel is not prohibited by this act.—*As amended May 23, 1908; Laws of 1908, vol. 2, ch. 486, pp. 1704-1705. See Bul. 112, pt. 2, p. 21.*

SEC. 188. *"Barrel" defined.* The term "barrel" when used in transactions of purchase or sale of apples, pears or quinces shall represent a quantity equal to one hundred quarts of grain or dry measure and shall be of the following dimensions: head diameter, seventeen and one-eighth inches; length of stave, twenty-eight and one-half inches; bulge, not less than sixty-four inches outside measurement. If the barrel shall be made straight, or without a bulge, it shall contain the same number of cubic inches as the barrel above described. Any person or persons making, manufacturing or causing to be made or manufactured barrels for use in the purchase or sale of apples, pears or quinces, or any person or persons packing apples, pears or quinces in barrels for sale or selling apples, pears or quinces in barrels containing a less quantity than the barrel herein specified shall brand said barrels upon each end and upon the outside, conspicuously, in letters one and one-half inches in length with the words, "short barrel."—*As added May 23, 1908; Laws of 1908, vol. 2, ch. 486, p. 1705. See Bul. 69, Rev., pt. 5, p. 428.*

Laws of 1893, ch. 338; Cumming and Gilbert's General Laws and other General Statutes, Supplement 1904, vol. 4, art. 13, p. 45.

NORTH CAROLINA.

GENERAL FOOD LAW.

SEC. 6. *Colors and preservatives prohibited; benzoic and sulphurous acids excepted.* For the purpose of this act an article of food shall be deemed adulterated * * *

Sixth. * * *

If it contain any of the following substances, which are hereby declared deleterious and dangerous to health when added to human food, to-wit: Colors which contain antimony, arsenic, barium, lead, cadmium, chromium, copper, mercury, uranium or zinc; or the following colors: gamboge, corallin, picric acid, aniline, or any of the coal-tar dyes; dulcin, glucin or any other artificially or synthetically prepared substitute for sugar except saccharine; paraffine, formaldehyde, beta-naphthol, abrastol, benzoic acid or benzoates, salicylic acid or salicylates, boric acid or borates, sulphurous acid or sulphites, hydrofluoric or any fluorine compounds, sulphuric acid or potassium sulphate or wood alcohol; *Provided*, that catsups and condimental sauces may, when the fact is plainly and legibly stated in the English language on the wrapper and label of the package in which it is retailed, contain not to exceed two-tenths of one per cent. of benzoic acid or its equivalent in sodium benzoate. Fermented liquors may contain not to exceed two-tenths of one per cent. of combined sulphuric acid, and not to exceed eight-thousandths of one per cent. of sulphurous acid.—*As amended February 1, 1908; Public Laws Extra Session 1908, ch. 117, pp. 130-131. See Bul. 69, Rev., pt. 5, p. 437.*

Approved April 13, 1899. Public Laws 1899, ch. 86, p. 216.

OHIO.

GENERAL FOOD LAWS.

SEC. 1. Adulteration and misbranding prohibited. That no person shall, within this state, manufacture for sale, offer for sale, sell, deliver or have in his possession with intent to sell or deliver any drug or article of food which is adulterated, within the meaning of this act; that no person shall, within this state, offer for sale, sell, deliver or have in his possession with intent to sell or deliver any drug or article of food which is misbranded, within the meaning of this act.—*As amended May 1, 1908; Laws of 1908 (Senate Bill No. 414), p. 257. See Bul. 69, Rev., pt. 6, p. 459.*

SEC. 3. Adulteration defined. An article shall be deemed to be adulterated within the meaning of this act:

(a) In the case of drugs: * * *

(b) In the case of food, drink, flavoring extract, confectionery or condiment: (1) If any substance or substances have been mixed with it, so as to lower or depreciate or injuriously affect its quality, strength or purity; (2) if any inferior or cheaper substance or substances have been substituted wholly, or in part, for it; (3) if any valuable or necessary constituent or ingredient has been wholly, or in part, abstracted from it; (4) if it is an imitation of, or is sold under the name of another article; (5) if it consists wholly, or in part, of a diseased, decomposed, putrid, infected, tainted or rotten animal or vegetable substance or article, whether manufactured or not or, in the case of milk, if it is the produce of a diseased animal; (6) if it is colored, coated, polished or powdered, whereby damage or inferiority is concealed, or if by any means it is made to appear better or of greater value than it really is; (7) if it contains any added substance or ingredient which is poisonous or injurious to health; (8) if, when sold under or by a name recognized in the eight decennial revision of the United States pharmacopoeia, or the third edition of the National Formulary, it differs from the standard of strength, quality or purity laid down therein; (9) if, when sold under or by a name not recognized in the eighth decennial revision of the United States pharmacopoeia, or the third edition of the National Formulary, but is found in some other pharmacopoeia, or other standard work on materia medica, it differs materially from the standard of strength, quality or purity laid down in such work; (10) if the strength, quality or purity falls below the professed standard under which it is sold; (11) if it contains any methyl or wood alcohol.—*As amended May 1, 1908; Laws of 1908 (Senate Bill No. 414), pp. 257-8. See Bul. 69, Rev., pt. 6, pp. 459-60.*

SEC. 3a. Misbranding defined. An article shall be deemed to be misbranded within the meaning of this act:

(a) In the case of drugs: * * *

(b) In the case of food, drink, flavoring extracts, confectionery or condiment: (1) If the package fails to bear a statement on the label of the quantity or proportion of any morphine, opium, cocaine, heroine, alpha or beta eucaine, chloroform, cannabis indica, chloral hydrate or acetanilide, or any derivative or preparation of any such substances contained therein; (2) if it be labeled or branded so as to deceive or mislead the purchaser, or purport to be a foreign product when not so; (3) if in package form, and the contents are stated in terms of weight or measure, they are not plainly and correctly stated on the

outside of the package; (4) in case of any flavoring extract, for which no standard exists, if the same is not labeled "artificial" or "imitation" and the formula printed in the same manner hereinafter provided for the labeling of "compounds" or "mixtures" and their formulae; (5) if the package containing it or any label thereon shall bear any statement, design or device regarding it or the ingredients or substances contained therein, which shall be false or misleading in any particular; Provided, that the provision of this act shall not apply to mixtures or compounds recognized as ordinary articles or ingredients of articles of food or drink, if each and every package sold or offered for sale be distinctly labeled in words of the English language as mixtures or compounds, with the name and percentage, in terms of 100 per cent., of each ingredient therein. The word "compound" or "mixture" shall be printed in letters and figures not smaller in either height or width than one-half the largest letter upon any label on the package and the formula shall be printed in letters and not smaller in either height or width than one-fourth the largest upon any label on the package and such compound or mixture must not contain any ingredient that is poisonous or injurious to health.—*Added May 1, 1908; Laws of 1908 (Senate Bill No. 414), pp. 258-9. See Bul. 69, Rev., pt. 6, p. 460.*

SEC. 5. Penalties. Whoever refuses to comply, upon demand, with the requirements of section 4, and whoever violates any of the provisions of this act, shall be fined not exceeding one hundred nor less than twenty-five dollars, for the first offense, and for each subsequent offense shall be fined not exceeding two hundred dollars nor less than one hundred dollars, or imprisoned in the county jail not exceeding one hundred, nor less than thirty days, or both. And any person found guilty of manufacturing, offering for sale or selling an adulterated article of food or drug under the provisions of this act, shall be adjudged to pay in addition to the penalties hereinbefore provided for, all necessary costs and expenses incurred in inspecting and analyzing such adulterated articles of which said person may have been found guilty of manufacturing, selling or offering for sale.—*As amended May 1, 1908. Laws of 1908 (Senate Bill No. 414), p. 259. See Bul. 69, Rev., pt. 6, p. 460.*

Passed March 20, 1884. 81 O. L., 67; Laning's Revised Statutes and Recodified Laws, 1905, title 5, ch. 8, pp. 1477-78.

SEC. 3a. Hindering inspector; penalty. Any person or persons who refuse to allow said commissioner, or any assistant commissioner or any inspector, or any of his agents entrance to any creamery, factory, store, salesroom, drug store, laboratory, booth, vehicle, steam or electric cars, or place which he desires to enter in the discharge of his official duty; or in any manner interfere with said commissioner, or any assistant commissioner, or any inspector, or agent in the discharge of his official duty; or refuse to deliver to him a sample of any article of food, drug, or linseed oil made, sold, offered or exposed for sale by such person or persons, when the same is requested and when the value thereof is tendered, shall be fined not exceeding two hundred nor less than fifty dollars, for the first offense, and for each subsequent offense shall be fined not exceeding three hundred nor less than one hundred dollars, or imprisoned in the county jail not exceeding one hundred, nor less than thirty days, or both.—*Added May 9, 1908; Laws of 1908 (Senate Bill No. 542), p. 386. See Bul. 69, Rev., pt. 6, p. 461.*

Laning's Revised Statutes and Recodified Laws, 1905, vol. 1, title 3, ch. 22, pp. 193-94.

DAIRY PRODUCTS.

SEC. 1. *Renovated butter must be so marked.* No person, firm or corporation shall manufacture for sale, offer or expose for sale, sell, exchange or deliver, or have in his possession with the intent to sell, exchange or deliver, any butter that is produced by taking original packing stock butter or other butter, or both, melting the same so that the butter fat can be drawn off or extracted, mixing the said butter fat with skimmed milk, or milk or cream, or other milk product, and rechurning or reworking the said mixture; nor shall any person, firm or corporation manufacture for sale, offer or expose for sale, sell, exchange or deliver, or have in his possession for any such purpose any butter which has been subjected to any process by which it is melted, clarified or refined, and made to resemble butter, and is commonly known as *bolled*, or cold extracted process or renovated butter, and which for the purpose of this act is hereby designated as "renovated" or "process butter," unless the same shall be branded or marked as provided in section two of this act.

SEC. 2. *Style of label prescribed.* Whoever, himself or by his agent, or as the servant or agent of another person shall sell, expose for sale or have in his custody or possession with intent to sell any "renovated" or "process butter," as defined in section one of this act, shall have the words "renovated butter" or "process butter" conspicuously stamped, labeled or marked in one or two lines and in plain Gothic letters, at least three-eighths of an inch square, so that the words cannot be easily defaced, upon two sides of each and every tub, firkin, box or package containing said "renovated" or "process butter," or, if such butter is exposed for sale uncovered or not in a case or package, a placard containing said words in the same form as above described in this section shall be attached to the mass in such a manner as to be easily seen and read by the purchaser. When "renovated" or "process butter" is sold from such package or otherwise at retail, in print, roll or other form, before being delivered to the purchaser, it shall be wrapped in wrappers plainly stamped on the outside thereof with the words "renovated butter," or "process butter" printed or stamped thereon in one or two lines, and in plain Gothic letters at least three-eighths of an inch square, and such wrapper shall contain no other words or printing thereon and said words "renovated butter" or "process butter" so stamped or printed on the said wrapper shall not be in any manner concealed, but shall be in plain view of the purchaser at the time of the purchase.

SEC. 3. *Penalty.* Any one violating any of the provisions of this act shall for a first offense be punished by a fine of not less than fifty nor more than two hundred dollars; for a second offense by a fine of not less than one hundred nor more than three hundred dollars or by imprisonment in the county jail or workhouse for not less than thirty days nor more than sixty days, or both.

SEC. 4. *Effect.* This act shall take effect sixty days after its passage.

Approved April 30, 1908. Laws of 1908 [Senate Bill No. 478], pp. 243-4.

SEC. 1. *Adulterated or watered milk; penalties.* That whoever by himself or by his servant or agent, or as the servant or agent of any other person, sells, exchanges or delivers, or has in his custody or possession with intent to sell or exchange or exposes or offers for sale or exchange adulterated milk, or milk to which water or any foreign substance has been added, or milk from cows fed on wet distillery waste, or starch waste, or from cows kept in a dairy or place which has been declared to be in an unclean or unsanitary condition by certificate of any duly constituted board of health or duly qualified health officer, within the county in which said dairy is located, or from diseased or sick cows, shall for a first offense, be punished by a fine of not less than fifty nor

more than two hundred dollars; for a second offense, by a fine of not less than one hundred dollars nor more than three hundred dollars, or by imprisonment in the jail or workhouse for not less than thirty nor more than sixty days; and for a subsequent offense, by fine of fifty dollars, and by imprisonment in the jail or workhouse for not less than sixty nor more than ninety days.—*As amended April 30, 1908; Laws of 1908 [Senate Bill No. 359], pp. 239-40. See Bul. 69, Rev., pt. 6, p. 472.*

Passed April 10, 1889, 86 O. L., 229; Laning's Revised Statutes and Recodified Laws, 1905, vol. 1, title 5, ch. 8, p. 1482.

SEC. 1. Refilling of milk containers. It shall be unlawful to fill or refill, with milk, cream or other milk product, any glass jar or bottle having the name of any person, firm or corporation blown therein, with intent to sell or vend such milk, cream or other milk product, provided, that the provisions of this section shall not extend to the person, firm or corporation whose name is blown in such glass jar or bottle, or a duly authorized agent or employe thereof.

SEC. 2. Sterilization of milk containers. It shall be unlawful to fill or refill, with milk, cream or other milk product, any glass jar or bottle with intent to sell or vend such milk, cream or other milk product, unless such glass jar or bottle be first thoroughly cleansed and sterilized.

SEC. 3. Penalty. Any person or persons guilty of violating the provisions of the preceding section of this act shall be fined not more than one hundred dollars.

Approved May 9, 1908. Laws of 1908 (House Bill No. 901), p. 454.

VINEGAR.

SEC. 1. Cider or apple vinegar defined. That no person shall manufacture for sale, offer, or expose for sale; sell or deliver, or have in his possession with intent to sell or deliver, any vinegar not in compliance with the provisions of this act. Any vinegar manufactured for sale, offered for sale, exposed for sale, sold or delivered, or in the possession of any person with intent to sell or deliver, under the name of cider vinegar, or apple vinegar, or any compounding of the word "cider" or "apple" as the name or part of the name of any vinegar, shall be the product made by the alcoholic and subsequent acetous fermentations of the juice of apples, shall contain no foreign substance, drugs or acids, is lævotatory, and shall contain not less than four (4) grams of acetic acid, not less than 1.6 grams of apple solids, of which not more than fifty (50) per cent. are reducing sugars, and not less than twenty-five hundredths (0.25) grams of apple ash in one hundred cubic centimeters (at a temperature of twenty [20] degrees centigrade); and the water-soluble ash from one hundred (100) cubic centimeters (at a temperature of [20] degrees centigrade) of the vinegar shall contain not less than ten (10) milligrams of phosphoric acid (P_2O_5), and which shall require not less than thirty (30) cubic centimeters of decinormal acid to neutralize its alkalinity.

(2) **Wine or grape vinegar defined.** Any vinegar manufactured for sale, offered for sale, exposed for sale, sold or delivered or in the possession of any person with intent to sell or deliver, under the name of wine vinegar, or grape vinegar, shall be the product made by the alcoholic and subsequent acetous fermentations of the juice of grapes, and shall contain, in one hundred (100) cubic centimeters (at a temperature of twenty [20] degrees centigrade), not less than four (4) grams of acetic acid, not less than one (1.0) gram of grape solids, and not less than thirteen hundredths (0.13) grams of grape ash.

(3) *Malt vinegar defined.* Any vinegar manufactured for sale, offered for sale, exposed for sale, sold or delivered or in the possession of any person with intent to sell or deliver, under the name of malt vinegar shall be the product made by the alcoholic and subsequent acetous fermentations, without distillation, of an infusion of barley malt or cereals whose starch has been converted by malt, is dextrorotatory, and shall contain in one hundred (100) cubic centimeters (at a temperature of twenty [20] degrees centigrade), not less than four (4) grams of acetic acid, not less than two (2) grams of solids, and not less than two-tenths (0.2) grams of ash; and the water-soluble ash from one hundred (100) cubic centimeters (at a temperature of twenty [20] degrees centigrade), of the vinegar shall contain not less than nine (9) milligrams of phosphoric acid (P_2O_5) and which shall require not less than four (4) cubic centimeters of decinormal acid to neutralize its alkalinity.

(4) *Distilled vinegar defined.* Any vinegar manufactured for sale, offered for sale, exposed for sale, sold or delivered or in the possession of any person with intent to sell or deliver, under the name of distilled vinegar, shall be the product made wholly or in part by the acetous fermentation of dilute distilled alcohol, and shall contain in one hundred (100) cubic centimeters (at a temperature of twenty [20] degrees centigrade), not less than four (4) grams of acetic acid, and shall be free from coloring matter, added during, or after distillation, and from coloring other than that imparted to it by distillation.—*As amended February 28, 1908; Laws of 1908 [Amended House Bill No. 931], pp. 28-9. See Bul. 69, Rev., pt. 6, p. 488.*

SEC. 2. *Labeling of fermented and distilled vinegars; other fermented vinegars; requirements.* All vinegar made by fermentation and oxidation without the intervention of distillation shall be branded "fermented vinegar," with the name of the fruit or substance from which the same is made. And all vinegar made wholly or in part from distilled liquor shall be branded "distilled vinegar," and all such distilled vinegar shall be free from coloring matter added during or after distillation and from color other than that imparted to it by distillation. And all fermented vinegar not otherwise provided for in said section 1, and not being distilled vinegar as defined in said section 1, shall contain not less than two (2) per cent. by weight, upon full evaporation (at the temperature of boiling water) of solids, contained in the fruit or grain or substance from which said vinegar is fermented, and said vinegar shall contain not less than two-and-a-half-tenths of one per cent. ash or mineral matter, the same being the product of the material from which said vinegar is manufactured. And all vinegar shall be made wholly from the fruit or grain from which it purports to be or is represented to be made, and shall contain no foreign substance, and shall contain not less than four per cent., by weight of absolute acetic acid.—*As amended February 28, 1908; Laws of 1908 [Amended House Bill No. 931], p. 29.*

Laning's Revised Statutes and Recodified Laws, 1905, vol. 1, title 5, ch. 8, p. 1499.

OKLAHOMA.

GENERAL FOOD LAWS.

SEC. 1. *Personnel of food commission.* A pure food, dairy and drug commission for the State of Oklahoma is hereby created, which shall be composed of the president of the State Board of Agriculture, the secretary of the State Board of Agriculture, the treasurer of the State Board of Agriculture, the State Commissioner of Health and the secretary of the State Board of Pharmacy.

SEC. 2. *Officers of the commission.* The president of said commission shall be the president of the State Board of Agriculture; the secretary of said commission shall be the State Commissioner of Health, and the treasurer of said commission shall be the treasurer of the State Board of Agriculture.

SEC. 3. *Powers and duties of commission; report.* It shall be the duty of said commission to carry into effect the provisions of this Act, and all other Acts in force or which may be hereafter enacted relating to foods, drugs and dairy products, and said commission is hereby authorized and empowered to promulgate and enforce such rules and regulations as they may deem proper and necessary to amend, alter and abolish the same from time to time not inconsistent with the provisions of this Act. They shall also have the power to appoint one dairy inspector, one food inspector, and one drug inspector, to prescribe their duties and powers, and to fix their compensation as hereinafter provided. Said commission shall make an annual report to the Governor on or about the first day of November of each year, giving in a concise manner, in said report, a full statement of the work of said commission, and accounting for all receipts and disbursements of the commission. Said commission shall be authorized and empowered to print their rules, regulations and announcements from time to time as they may deem necessary. The annual report of said commission shall be printed, published and distributed the same as reports of other State commissions. Said commission shall have authority to lease, rent and contract for such office or offices as they may deem necessary for the convenient transaction of the business of said commission at the seat of the State government.

SEC. 4. *Duties of officers.* The president of the commission shall preside at all meetings of the commission and perform such other duties as the commission by their rules may prescribe.

The secretary of the commission shall keep a record of all proceedings of the commission and perform such other duties as are prescribed in this Act, or which may be prescribed by said commission. He shall keep an accurate account of the expenses of said commission and file monthly itemized statements of such expenses with the State Auditor. He shall receive all moneys collected by said commission, and promptly pay the same to the treasurer of said commission, taking a duplicate receipt therefor, one of which shall be filed with the State Auditor, and the other retained by said secretary. He shall make, on the first day of each month, a report to the Governor, covering the entire work of said commission for the preceding month and show, among other things, the number of manufactureries^a and other places inspected, and by whom; the number of specimens of food articles analyzed, and a list of cases

^a So in Statutes.

in which adulteration was found, the number of complaints entered against persons for the violation of the law relative to the adulteration of articles named in this Act; the number of convictions had and the amount of fines imposed and collected and sentences passed; and it shall be his duty to cause prosecution to be made against parties violating the provisions of this Act. During the adjournment of the commission the secretary shall be authorized and empowered to carry on the work of the commission.

It shall be the duty of the treasurer to receive, receipt for, and safely keep all funds coming into his hands, and to deposit the same with the State Treasurer at least once each month, taking his receipt therefor, and make to the president of the commission on the first day of each month a full statement of the receipts and disbursements of his office for the month next preceding.

The members of said commission shall make and subscribe to the same oath of office as that prescribed in the Constitution of the State of Oklahoma for other officials, and the secretary and treasurer shall each give bond in the sum of five thousand dollars each for the faithful performance of the duties of their respective offices, which bond shall be approved by the Governor and filed with the Secretary of State.

SEC. 5. Compensation. The said board of commissioners shall receive their actual expenses while engaged in the performance of their duties in connection with this Act. They are hereby authorized to employ a stenographer or clerk at a salary not to exceed seventy-five dollars per month, also to fix the compensation of the inspectors, not to exceed three dollars per day and actual expenses.

SEC. 6. State laboratories for analysis of samples. For the purpose of this Act there is hereby established two State laboratories for the analysis of food, feeding stuffs, drugs and medicines, which shall be under the supervision of said commission. One of said laboratories shall be established and located at the State University, and the director of said laboratory shall be the professor of the department of chemistry in the State University. The other laboratory shall be established at the State Agricultural and Mechanical College at Stillwater, and the director of said laboratory shall be the chemist of the experiment station in the said Agricultural and Mechanical College. To the said laboratory at the State University all samples of drugs and medicines shall be sent for analysis and examination. And to the said laboratory at the said Agricultural and Mechanical College shall be sent for analysis and examination all samples of foods and feeding stuffs, and all samples of dairy products. The said University and the said Agricultural and Mechanical College shall employ such additional chemists and assistants as are necessary properly and expeditiously to examine and analyze such drugs, medicines, food and dairy products as are sent them by the said commission for the purpose of determining whether such articles are adulterated, misbranded and mislabeled within the meaning of this Act, and if it shall appear that any of such specimens are adulterated, mislabeled or misbranded within the meaning of this Act, the secretary of the commission shall at once certify the facts to the county attorney of the county in which such sample was taken, with a copy of the results of the analysis of the examination of such samples, duly authenticated by the analyst or officer making such examination or analysis, under oath of such analyst or such officer; Provided, that said commission may submit to the department of chemistry at the said State University or at the said experiment station of Agricultural and Mechanical College, any sample or samples of any article of food, drugs, medicines or dairy product for analysis, and the directors of such departments shall make and furnish the commission such analysis or analyses.

The said commission, out of the appropriation hereinafter provided, may employ and fix the compensation of other and additional clerical and professional assistants.

SEC. 7. *Inspection and prosecution.* Said pure food commission is hereby given full jurisdiction over the regulation and control of the manufacture and sale of all foods, drugs and medicines and dairy products, and shall be authorized and empowered to make inspections concerning the purity of the same and to bring prosecutions for violations as provided herein in the case of foods, drugs and dairy products, and shall exercise the necessary police authority in the enforcement of this Act for the preservation of the public health.

SEC. 8. *Adulteration and misbranding prohibited.* The manufacture, production, preparation, compounding, packing, selling, offering or keeping for sale within the State of Oklahoma, or the introduction into the State from any other State or Territory, or the District of Columbia, or from any foreign country of any article of food or dairy product which is adulterated, mislabeled or misbranded within the meaning of this Act is hereby prohibited.

Any person, firm, company or corporation who shall import or receive from any other State, Territory, or the District of Columbia, or from any foreign country, or who having so received, shall deliver, for pay or otherwise, or offer to deliver to any other person any article of food or dairy product mislabeled or misbranded within the meaning of this Act, or any person, firm or corporation who shall manufacture or produce, prepare, compound, pack or sell or offer or keep for sale in the State of Oklahoma any such adulterated, mislabeled or misbranded food or dairy product shall be guilty of a misdemeanor: Provided, that no article of food or dairy product shall be deemed adulterated, mislabeled or misbranded within the provisions of this Act, where prepared for export beyond the jurisdiction of the United States and prepared or packed according to the specifications or directions of the foreign purchaser, when no substance is used in the preparation or packing thereof in conflict with the laws of the foreign country to which said article is intended to be shipped.

SEC. 9. *Term "person" defined.* The word person, as used in this Act, shall be construed to impart the singular and the plural, as the case may demand, and shall include corporations, companies, societies and associations. When construing and enforcing the provisions of this Act, the act, omission or failure of any officer, agent or other person acting for or employed by any corporation, company, society or association, within the scope of employment of his office, shall in every case be also deemed to be the act, omission or failure of such corporation, company, society or association, as well as that of the person.

SEC. 10. *"Food" and "dairy products" defined.* The term "food," as used in this Act, shall include all articles of food, drink, liquor, beverage, confectionery or condiment used by man or other animal, whether simple, mixed or compound. The term "dairy product," as used in this Act, shall include milk, cream, butter, cheese, skimmed milk, buttermilk or any modification of the foregoing materials or compounds containing one or more of same and all products derived from milk.

SEC. 11. *Food standards.* The standard of purity of foods shall be that proclaimed by the Secretary of the Department of Agriculture of the United States.

SEC. 28. *Food adulteration defined.* Food shall be deemed to be adulterated within the meaning of this Act in any of the following cases:

First: If any substance has been mixed or packed with the food so as to reduce or lower or injuriously affect its quality, purity, strength or food value.

Second: If any substance has been substituted wholly or in part for the article of food.

Third: If any essential or valuable constituent or ingredient of the article of food has been wholly or partly abstracted.

Fourth: If it be mixed, colored, powdered, coated or stained in any manner whereby damage or inferiority is concealed.

Fifth: If it contain any added poisonous or other added deleterious ingredient in the food.

Sixth: If it consists in whole or in part of a filthy, decomposed or putrid animal or vegetable substance, or any portion of an animal or vegetable unfit for food, whether manufactured or not, or if it is the product of a diseased animal, or one that has died otherwise than by slaughter.

SEC. 29. *Misbranding of food defined.* Food shall be deemed mislabeled or misbranded within the meaning of this Act in any of the following cases:

First: If it be in imitation of or offered for sale under the distinctive name of another article of food.

Second: If it be labeled, or branded, or colored so as to mislead or deceive the purchaser, or if it be falsely labeled in any respect, or if it purport to be a foreign product when not so, or if the contents of the package as originally put up shall have been removed in whole or in part and other contents shall have been placed in such package.

Third: If in package form and the contents stated in terms of weight or measure, they are not plainly and correctly stated on the outside of the package.

Fourth: If the package containing it or its label shall bear any statement, design or device regarding the ingredients or the substance contained therein, which statement, design or device shall be false or misleading in any particular.

Fifth: When the package bears the name of the manufacturer, jobber or seller, or the grade of the product, it must bear the name of the real manufacturer, jobber or seller, and the true grade or class of the product, the same to be expressed in clear, distinct English words in legible type; Provided, that an article of food shall not be deemed misbranded if it be a well known food product of a nature, quality and appearance, and so exposed to public inspection as not to mislead or deceive or tend to mislead or deceive a purchaser, and not misbranded and not of the character included within the definitions of one to four of this section; Provided, that all packages of imitation butter and cheese shall be so labeled.

SEC. 30. *License for selling proprietary preparations.* * * * Before any manufacturer or proprietor of any food, proprietary or secret preparation, or product of any food or article used in the preparation of food, drug or liquor, or medicine, shall sell, expose or offer for sale or exchange within said State, he shall first procure from the said commission a license or permit to sell the same, and shall pay a filing fee, and for each license or permit so filed in any sum not to exceed \$30.00, as required by said commission, said filing fee to be paid annually.

[Secs. 31-34 relate to drugs.]

SEC. 35. *Misbranding defined.* That the term "misbranded," as used herein, shall apply to all articles which enter into the composition of foods and drugs, the package or label of which shall bear any statement, design or device regarding such article or the ingredients or substances contained therein, which shall be false or misleading in any particular.

SEC. 36. *"Package" defined.* The term "package," as used in this Act, shall be construed to include the original unbroken package, phial, bottle, jar, demi-john, carton, bag, case, can, box, barrel, or any receptacle, vessel or container of whatsoever material or nature which may be used by a manufacturer, pro-

ducer, jobber, packer or dealer for enclosing any article of food or any drug or medicine when exposed or offered for sale.

SEC. 37. Possession evidence of violation of act. The possession of any adulterated, mislabeled or misbranded article of food, dairy product or drug, or the offering for sale or the sale of any adulterated, mislabeled or misbranded food, dairy product or drug, by any manufacturer, producer, jobber, packer or dealer in food or drugs, or broker or commission merchant, agent, employee or servant of any such manufacturer, producer, jobber, packer or dealer, shall be prima facie evidence of the violation of this Act.

SEC. 38. Hotel signs for imitation butter and cheese, adulterated milk and lard. Whenever any hotel, tavern, restaurant, or boarding house shall knowingly serve for the use of their patrons such food as is defined in this Act as compounds, imitations, blends, renovated butter, imitation cheese, adulterated milk or adulterated lard [they] shall keep conspicuously posted or printed in a bill of fare a list of the articles of food so served in plain and legible words, the brands or labels upon the original package or the constituent parts of such food articles.

SEC. 44. Imitation honey must be so labeled. It shall be unlawful for any person to sell, offer, or expose for sale or exchange, any honey which has not been wholly made by bees, unless the same is labelled "imitation" and contains nothing that is injurious to health.

SEC. 47. Protection of meat and game. Every dealer or peddler in slaughtered fresh meats, fish, fowl or game for human food, at wholesale or retail in the transportation of such food from place to place, to customers, shall protect the same from dust, flies and other vermin, or substances which may injuriously affect it by securely covering it while being so transported.

SEC. 48. "Sale" defined. The taking of orders or the making of agreements or contracts by any person, firm or corporation, or by an agent or representative thereof, for the future delivery of any of the articles, products, goods, wares or merchandise embraced within the provisions of this Act, shall be deemed a sale within the meaning of this Act.

SEC. 49. Penalty for misbranding or defacing label. Whoever shall falsely brand, mark, stencil or label any article or product required by this Act to be branded, marked, stenciled or labeled, or shall remove, alter or deface, mutilate, obliterate, imitate, or counterfeited any brand, mark, stencil, or label so required, shall be deemed guilty of a misdemeanor, and upon conviction thereof shall be punished by a fine of not less than fifty dollars nor more than five hundred dollars, or by imprisonment in the county jail for not less than six months nor more than one year, or by both such fine and imprisonment, for each and every offense.

SEC. 51. Penalty. Whoever shall do any of the acts or things prohibited or willfully neglect or refuse to do any of the acts or things enjoined by this Act, or in any way violate any of its provisions, shall be deemed guilty of a misdemeanor, and where no specific penalty is prescribed by this Act, shall be punished by a fine of not less than twenty-five nor more than five hundred dollars or by imprisonment in the county jail for a period of not less than thirty days nor more than ninety days, or by both such fine and imprisonment.

SEC. 55. Colored distilled vinegar illegal. It shall be unlawful for any person, firm, or corporation to sell or offer for sale in this State, any colored, distilled vinegar.

SEC. 58. Possession shows intent to commit offense. If any person shall have in his possession or control any article or articles of adulterated or misbranded or mislabeled food, drugs, or medicines, contrary to the provisions of this Act, he shall be held to have possession of property with intent to use it as a means of

committing a public offense, and all the provisions of the chapter in the statutes of the State of Oklahoma relating to search warrants and proceedings thereby shall apply.

SEC. 59. Appropriation. There is hereby appropriated out of the funds in the state treasury not otherwise appropriated, the sum of five thousand dollars, or so much thereof as may be necessary for the purpose of paying the salaries and expenses of the officers created under this Act, and for the maintenance of the State Laboratories created under this Act, and the necessary expenses incurred in the enforcement of this Act.

SEC. 60. Duties and powers of food inspectors; sheriffs appointed agents; sampling. It shall be the duty of the pure food inspectors to make, or cause to be made, by one of the directors of the state laboratories examinations and analyses of foods or drugs on sale in Oklahoma, suspected of being adulterated, mislabeled, misbranded, impure or unwholesome, in contravention of the law. And if upon examination or analysis, it is found that said food or drug is adulterated, mislabeled, misbranded, impure or unwholesome, it shall be the duty of the pure food inspector to make complaint against the manufacturer or vendor thereof in the proper county and to furnish the evidence thereon, and thereof to obtain a conviction of the offense charged. And the sheriffs of the respective counties of the State are hereby appointed and constituted agents for the enforcement of this Act, and the pure food inspector or any sheriff shall have free access at all reasonable hours for the purpose of examining any place where it is suspected that any article of adulterated, mislabeled, misbranded, impure or unwholesome food, medicine or drug exists, and such food inspector or sheriff, upon tendering the market price of such article, if a sale be refused, may take from any person, firm or corporation, samples of any article suspected of being adulterated, mislabeled, misbranded, impure or unwholesome, for the purpose of examination or analysis, and divide the said article into three parts, and each part shall be sealed by the pure food inspector or sheriff seizing the said article, with a seal provided for that purpose. If the package be less than four pounds or in volume less than two quarts, three packages of approximately the same size shall be purchased and the marks and tags upon each package noted as above. One shall be delivered to the party from whom purchased, or the party guaranteeing such merchandise, one sample shall be sent or delivered to one of the directors of the state laboratories for examination and analysis, and the third shall be held by the sheriff of the county in which said article was seized, under his seal, for future reference should the case come to trial.

SEC. 61. Sheriff's fees and expenditures. For his services hereunder, the sheriff shall be allowed the same fee for travel allowed by law to sheriffs on service of criminal process, together with such compensation as by the board of county commissioners of his county, may be deemed reasonable, and all amounts expended by him in procuring and transmitting the said samples, which fees and amount expended shall be audited and allowed by said board of county commissioners and paid by said county as other bills of said sheriff.

SEC. 62. Prosecution. It shall be the duty of all prosecuting officers of the State to prosecute to completion all suits brought under the provisions of this Act, upon the complaint of any member of the pure food, dairy and drug commission or any other citizen of the State of Oklahoma. It shall be the duty of all city and county health officers to take cognizance of and to report all prosecutions or violations of this Act, which may be brought to their notice or they have cognizance of within their jurisdiction.

SEC. 63. Disposition of fines. One half of all fines collected by any court or judge for the violation of the provisions of this Act, shall be paid to the state

treasurer, one half shall be paid into the treasury of the county where such cases are prosecuted.

SEC. 64. *Hindering inspectors a misdemeanor; penalty.* It shall be a misdemeanor for any person, firm or corporation to refuse to sell to the pure food inspector, sheriff or agent of the pure food, dairy and drug commission any sample of food or drug suspected of being adulterated, misbranded, mislabeled, impure or unwholesome, upon the tender of the market price thereof, or to conceal such food, liquor, drug or medicine from such officer, or to withhold from him information where such food or drug is kept or stored. Any such person so refusing to sell, or concealing such food, medicine, or drug or withholding such information from said officer upon conviction, shall be punished by a fine of not less than twenty-five dollars, nor more than one hundred dollars, or by imprisonment in the county jail for not less than thirty days nor more than ninety days.

SEC. 65. *Guarantee for protection of dealer.* No dealer shall be prosecuted under the provisions of this Act, when he can establish a guarantee signed by the wholesaler, jobber, manufacturer, or other party residing in the United States from whom he purchased such article to the effect that the same is not adulterated, mislabeled, or misbranded within the meaning of this Act. Said guarantee to afford protection must contain the name and addresses of the party or parties making the sales of such articles to said dealer, and an itemized statement showing the article purchased, or a general guarantee may be filed with the secretary of the United States department of Agriculture, by the manufacturer, wholesaler, jobber, or other party in the United States and given a serial number, which number shall appear on each and every package of goods, sold under such guarantee with the words "guaranteed under the food and drugs Act, June thirtieth, nineteen hundred six." In case the wholesaler, jobber, manufacturer, or other party making such guarantee to such dealer resides without this State, and it appears from the certificate of the director of the state laboratory that such article or articles were adulterated, mislabeled or misbranded, within the meaning of this Act or the "National Pure Food Act" approved June thirtieth, nineteen hundred six, the attorney general of this State must forthwith notify the attorney general of the United States of such violation.

SEC. 66. *Repeal.* All acts and parts of acts in conflict with this Act are hereby repealed.

SEC. 67. *Goods bought prior to passage of law exempt.* That in any prosecution for any violation of any provisions of this Act, relative to the manufacture, possession or sale of any alleged food product or drug, it shall be a valid defense for the defendant to prove that the articles described in the complaint were in his possession as a part of his stock in trade in this State prior to the time of the passage and approval of this Act.

SEC. 68. *Date of effect.* An emergency is hereby declared by reason whereof it is necessary for the immediate preservation of the public health, peace and safety, that this Act take effect from and after its passage and approval.

Approved May 26th, 1908. Session Laws 1907-1908, ch. 37, pp. 403-426.

BREAD.

SEC. 55. * * * *Labeling of bread as to time of baking.* It shall be unlawful for any person in this State to sell, or offer to sell any loaf bread, manufactured outside of the State of Oklahoma without having pasted on

each loaf of such bread, a label having written or printed thereon the date and hour of the day the same was baked, and it shall be unlawful to sell any bread over seventy-two hours after the same was baked, without informing each person purchasing or offering to purchase the same that it is "stale bread."

Session Laws 1907-1908, ch. 37, p. 422.

CONFECTIONERY.

SEC. 57. *Adulteration of confectionery; penalty.* Any person manufacturing for sale, or selling or offering to sell or exchange any candies, or confectioneries, adulterated by admixture of terra alba, barytes, talc, or other earthly^a or mineral substances, or any poisonous colors, flavors or extracts, or other deleterious ingredients detrimental to health, shall upon conviction thereof before a court of competent jurisdiction be punished by a fine of not less than ten nor more than one hundred dollars or by imprisonment in the county jail not less than ten days nor more than thirty days, or by both such fine and imprisonment.

Session Laws 1907-1908, ch. 37, p. 423.

DAIRY PRODUCTS.^b

SEC. 12. *Standards for milks and cream.* The following minimum standards of purity for milk and cream are hereby established: Milk shall contain not less than three per centum of butter fat, and cream contain not less than eighteen per centum of butter fat, and it is hereby made unlawful for any person or persons to sell or offer for sale in this State, except under test, any milk or cream falling below said minimum standard therefor. In no event shall milk or cream be sold or offered for sale when produced within thirty days before or fifteen days after calving.

In testing milk or cream for commercial purposes under the provisions of this Act, the same shall be done in accordance with the rules and regulations therefor prescribed by said commission.

All cream sold in the State of Oklahoma shall be tested for butter fat by the following prescribed method:

The Babcock test shall be employed, using a weighed sample of eighteen grams, weighed on a delicate balance and tested in a nine inch bottle, graduated to at least five-tenths per cent of the column of fat read above a temperature of one hundred thirty degrees Fahrenheit. It is hereby made unlawful for any person to test milk or cream at any milk or cream receiving station or at any place where milk or cream is tested for commercial purposes, without first securing a permit issued by said commission. Said commission is hereby authorized to issue to any person making application therefor, a permit to test milk or cream, if, on examination such person be found competent to test milk or cream; said examination shall be given under the direction of said commission at convenient places therefor throughout the State. All permits so issued shall expire on the thirtieth day of June next succeeding the date of issuance.

It is hereby made the duty of said commission to supply to each inspector, a tester under this Act, at the time of issuing to him a license or permit and copy of all rules and regulations formulated by said commission relating to the dairy industry then in force. It shall be unlawful for any owner or employe of any

^a So in Statutes.

^b See also General Food Laws, page 54.

creamery or cheese factory, or for any person, to improperly manipulate or under-read the Babcock test.

It shall be unlawful for any person, agent or employe of any creamery, cheese factory or person to so manipulate the sampling that the sample taken does not fairly represent the uniform mixture of the cream or milk from which it was taken.

If milk sold or offered for sale under the provisions of this Act as pure milk, is shown upon analysis by weight, to contain more than eighty-seven and five one-hundredths per centum of watery fluid, or to contain less than twelve and fifty one-hundredths per centum of milk solids, or less than three per centum of butter fat, or if the gravity at sixty degrees Fahrenheit is not between one hundred twenty-nine one-thousandths to one hundred thirty-three one-thousandths, it shall be deemed adulterated. If milks sold or offered for sale under the provisions of this Act as skimmed milk has a gravity at sixty degrees Fahrenheit less than one and thirty-two one-thousandths, and greater than one and thirty-seven one-thousandths, it shall be deemed to be adulterated.

SEC. 13. *Analysis of suspected milks.* Whenever the pure food inspector has reason to believe that any milk found by him is adulterated, he shall take specimens thereof and test the same with such instruments as are used for such purpose, and he shall make an analysis thereof, showing total solids, the percentage of butter, the percentage of water and the percentage of ash, and if the result of such test and analysis indicated that the milk has been adulterated or deprived of its fat below the requirements of section twelve of this Act, the same shall be prima facie evidence of such adulteration in a prosecution under this Act.

SEC. 14. *City milk inspectors.* Authority is hereby given the city council of any city, or the board of trustees of any town or village, to appoint an inspector of milk in any such city or town and to fix his compensation, and when appointed the said inspector of milk shall have all the powers given him by section twenty of this Act, and shall perform all the duties required of inspectors of milk as provided herein, and such other powers and duties as may be conferred or imposed by the ordinances of said cities or towns.

SEC. 15. *Adulteration of milk prohibited; milk defined.* No person shall offer for sale, sell, exchange or deliver or have in his possession with the intent to sell, exchange or deliver, any milk, to which water, chemicals or preservatives, or any other foreign substance has been added. The term "milk," as used in this Act, shall include all milk, cream or milk, in its natural state as drawn from the cow.

SEC. 16. *Adulteration of milk a misdemeanor.* Whoever shall adulterate, by himself, or by his servant or agent, or sell, exchange or deliver, or have in his custody or possession, with intent to sell or exchange the same, or expose or offer for sale, adulterated milk, or milk to which water or any foreign substance or substances in any state of fermentation or putrification or from any sick or diseased cows shall be guilty of a misdemeanor.

SEC. 17. *Skimmed milk regulations.* Whoever shall adulterate, or cause to be adulterated, sell, exchange or deliver, or have in his custody or possession, with intent to sell or exchange the same or expose or offer for sale or deliver as pure milk, any skimmed milk, from which the cream or any part thereof has been removed, shall be guilty of a misdemeanor.

SEC. 18. *Labeling of skimmed milk.* Any dealer in milk who shall, by himself, servant or agent, sell, exchange or deliver, or have in his custody or possession, with intent to sell, exchange or deliver the same, milk from which the fat has been removed, so as to reduce the same below the requirements of sec-

tion thirteen of this Act, unless in a conspicuous place above the center upon the outside of every vessel, can or package from which any such milk is sold, the words "skimmed milk" are distinctly painted or printed, shall be guilty of a misdemeanor.

SEC. 19. *Manufacturer and dealers in imitation cheese and butter defined; "creamery" and "cheese factory" defined.* Every person who in any manner produces imitation butter or imitation cheese shall be considered a manufacturer thereof.

Any person who sells imitation butter or imitation cheese in packages or quantities containing more than ten pounds, shall be deemed a wholesale dealer thereof.

Any person who deals in imitation butter or imitation cheese in packages containing less than ten pounds each, shall be deemed a retail dealer thereof.

The word "creamery," as used in this Act, is hereby defined as a factory where cream or milk from two or more dairy herds, with or without the addition of salt and coloring matter, is churned into butter. The term "cheese factory," as used in this Act, is hereby defined to be a factory where milk from two or more dairy herds, with or without the addition of salt and coloring matter, is manufactured into cheese. The term "to test milk or cream," as used in this Act, is hereby defined as the process or method by which the percentage of butter fat in said milk or cream is determined.

SEC. 20. *Permits for butter, cheese, and ice cream factories, etc.* It is hereby made unlawful for any manufacturer, wholesale dealer or retail dealer in imitation butter or imitation cheese, or both, to enter upon or engage in the business of producing, manufacturing, handling or selling imitation butter or imitation cheese without first procuring from said commission a permit describing the occupation and place of business of the person engaged in the same, which permit shall expire on the thirtieth day of June following its issuance unless sooner revoked. It is hereby made unlawful to operate any creamery or cheese factory, or both, without first securing from said commission a permit, in which permit shall be described the place of business of the applicant and the business to be conducted under said permit. It is hereby made unlawful for any person to engage in the business of buying cream for any butter, cheese or ice cream factory without first securing from said commission a permit, in which permit shall be described the place of business of the applicant and the business to be conducted under said permit. It is hereby made unlawful for any person engaged in the business of buying cream in this State for the purpose of manufacture or shipment out of the State to make discriminations in the price paid for such cream when purchased upon the same day at different places in said State.

SEC. 21. *Cost of permits.* For permit issued in connection with this Act, there shall be charged and collected annually as follows: From each manufacturer of imitation butter or imitation cheese, the sum of fifty dollars; for each wholesale dealer in imitation butter or imitation cheese, twenty-five dollars; from each retail dealer in imitation butter or imitation cheese, ten dollars; from each creamery or cheese factory, five dollars; from each person engaged in the testing of cream or milk for commercial purposes, one dollar; said fees shall be paid as provided by law, in advance of the issuance of any permit. All permits so issued shall expire on the thirtieth day of June next succeeding the date of issuance. When a permit is issued to such manufacturer, dealer, creamery or factory after the beginning of any license year, the fee charged and collected therefor shall be proportioned to the unexpired portions of such year, counting from the first day of the month in which such license is issued.

SEC. 22. *Unsanitary implements unlawful.* It is hereby made unlawful to use or employ, in and about the keeping or handling of any milk, cream or dairy products to be used as food, any pail, can, vessel, churn, separator or other implement which is in an unclean or unsanitary condition, or to operate any creamery or factory in the manufacture of any dairy products which is in an unclean condition.

SEC. 23. *Diseased cows.* It shall be unlawful knowingly to sell or offer for sale any milk or cream from diseased or unhealthy cows, or from cows kept in a filthy or unsanitary condition, and the pure food, dairy and drug commission is hereby empowered to adopt and promulgate such rules and regulations governing the use of diseased milch cows, and products derived from such cows, and to employ such scientific assistance in the enforcement of said rules and regulations.

SEC. 24. *Duties of chief dairy inspector.* The said chief dairy inspector shall act on all reports and complaints he may receive from the secretary of the commission and from owners or managers of creameries, cheese factories, farmers and others who are interested in dairy products, wherein are reported to him any violations of this Act, or conditions which result in making or rendering dairy products used or to be used for dairy, food or commercial purposes, unclean or unwholesome, and take such action thereon as may be directed by said commission or the secretary thereof and as may be permitted by this Act, or he may deem necessary and proper for improving and advancing the best interests of the dairy industry in this State and public health. He shall also, each month make to the commission a concise report of his transactions as said chief dairy inspector, and make such recommendations in the premises as he shall deem proper and for the better perfection and encouragement of said industry. It shall be the duty of said chief dairy inspector, and such other dairy inspectors as may be appointed by the commission from time to time, to inspect farm dairies, milk and cream receiving stations, creameries, factories and places where dairy products are produced, handled, tested, manufactured, sold or offered for sale and the products thereof, and all utensils, machinery, appliances, implements and methods used or employed in connection therewith.

SEC. 25. *Authority of inspector; sampling.* The said dairy inspector shall have full access, ingress and egress to and from all places where dairy products intended for sale are produced, manufactured, stored, transported, kept or offered for sale. They shall also have the power and authority to open any package, can or vessel containing such products, and may inspect the same and take true samples therefrom for analysis upon paying therefor the full value thereof to the party entitled thereto. Each sample so taken shall be divided into three parts, each equal to the other in amount and quality, two of said parts to be delivered to the chemist of said commission at the Agricultural and Mechanical College at Stillwater, the other sample so taken to be preserved in the office of the commission, and upon application delivered to the person or persons from whom taken when applied for by him, his agent or attorney, provided that said samples shall each be carefully sealed and labeled when and where taken. It shall be unlawful for any person or persons to obstruct, hinder or delay any of said inspectors in the discharge of their official duties.

SEC. 26. *Prosecution.* If it shall appear from the report of the chemist, report of said dairy inspectors, or any of them, that any of the provisions of this Act have been violated, the secretary shall certify the facts to the proper county attorney with a copy of the result of the analysis, if any has been made, duly authenticated to by the chemist under oath. It shall be the duty of every county attorney to whom the secretary shall report any violation of this Act, or any

other Acts relating to dairy products, to cause proceedings to be commenced in the name of the State of Oklahoma and prosecute the same without delay for the recovery of any fines and penalties in such cases provided.

SEC. 27. "*Cream check;*" *penalty.* It shall be the duty of every person engaged in the buying of cream for manufacture into butter, cheese, ice cream or other products, to give a receipt or "cream check" therefor, clearly and thoroughly stating the name and principal place of business of the person, firm, corporation or association for whom such cream is purchased, and any person, firm, corporation or association who shall violate any of the provisions of this Act shall be deemed guilty of a misdemeanor, and in addition thereto, shall forfeit his license or permit to engage in such business.

SEC. 56. *Each illegal sale of milk a separate offense; hindering inspector.* Each and every quantity of milk sold or exposed for sale or exchange contrary to the provisions of this Act, shall constitute a separate offense.

Any person who shall refuse to permit the pure food inspector, or his assistant, to perform his duty under this Act, either by refusing him entrance to his premises, or by concealing any milk or refusing to permit any animal or milk on premises wherein the animals are kept to be viewed and inspected as herein provided, or by in^a any manner hindering or resisting any said inspector or assistant inspector in the performance of his duty, shall be guilty of a misdemeanor.

Session Laws 1907-1908, ch. 37, pp. 408-423.

FLAVORING EXTRACTS.

SEC. 43. *Imitation flavoring extracts must be so labeled.* It shall be unlawful for any person to manufacture, sell or offer for sale or exchange as extracts, flavoring which was not made from the natural fruit unless the same are labelled "Imitation." Provided, that the word "Imitation" must immediately precede the name of the flavoring, in the same type and style. Such flavoring shall be free from coloring matter deleterious to health.

SEC. 45. *Vanilla extract.* It shall be unlawful for any person to manufacture, sell, offer or expose for sale, or exchange, extract of vanilla, essence of vanilla, or spirits of vanilla, not wholly made from the extracted matter of vanilla beans.

Session Laws 1907-1908, ch. 37, p. 420.

FLOUR.

SEC. 52. *Labeling of flour compounds.* Within this State no person shall manufacture, offer or expose for sale, keep in his possession with intent to sell or exchange, any flour made from wheat containing any products of corn, rice or other foreign substances, unless each and every package thereof be distinctly and legibly branded or labeled "flour compound" in letters not less than one-half inch in length and be followed with the name of the maker and mill and the location of such flouring mill.

SEC. 53. *Possession of flour compounds evidence of intent to sell.* The having possession of any "flour compound" or "meal compound" which is not branded or labelled as hereinbefore required and directed upon the part of any person engaged in the public or private sale of such article shall, for the purpose of this Act, be deemed prima facie evidence of intent to sell the same.

^a So in Statutes.

SEC. 54. "*Sale*" *defined*. The taking of orders or the making of agreements or contracts by any person, firm or corporation, or by an agent or representative thereof, for the future delivery of any "flour compound" or "meal compound" shall be deemed^a a sale within the meaning of this Act.

Session Laws 1907-1908, ch. 37, p. 422.

HONEY.

See General Food Laws, sec. 44, page 58.

LARD.

SEC. 39. *Exemptions; lard compound and substitute defined*. The provisions of this Act shall not apply to substances for sale in this state, made in the semblance of lard, if the ingredients or component parts shall consist of pure lard, beef fat or pure stearine and cottonseed oil that is one per cent of the legitimate and exclusive fat of the hog, or pure lard, pure stearine or beef fat, and ninety-nine per cent of cottonseed oil, and the tierce, tub, pail or package containing the same is distinctly and legibly branded, marked or labeled, "lard compound" or "compound lard" or "lard substitute" in letters proportional to the size of the package, and if such mixture contain any other substance than pure lard, pure stearine or beef fat, or pure cottonseed oil, then the person or corporation so manufacturing shall cause the tierce, barrel, tub, pail or package containing the same to be distinctly and legibly branded, marked or labeled "adulterated lard"^a the term "lard compound" or "compound lard," as used herein, shall include all articles of food used as lard, or made in the semblance of lard, which shall be composed of two or more ingredients or component parts, consisting of either cottonseed oil, pure lard or hog lard, beef fat or pure stearine, the percentage of either of the two or more ingredients used to be in the discretion of the manufacturer. The term "lard substitute," as used herein, shall apply to any compound which may consist of two or more of the aforesaid ingredients or of cottonseed oil alone. Neither shall the provisions of this Act apply to mixtures or compounds consisting of mixtures of beef suet, beef fat or pure stearine, and cottonseed oil, or of cottonseed oil alone, when said mixtures or compounds used as ordinary articles of food, or cooking "compounds" are manufactured and sold under their proper trademark, and when the tierce, barrel, tub, pail or package containing the same shall be distinctly and legibly branded or labeled with the name of the mixture or compound, in letters proportioned to the size of the package, and the name and location of the person, firm or corporation manufacturing the same.

SEC. 40. *Labeling of lard compounds, etc*. Every manufacturer, trader or dealer who, by himself or agent, or as the servant or agent of another person, offers or exposes for sale, or sells or exchanges any form of lard substitute or adulterated lard as hereinbefore defined, shall securely fix or cause to be affixed to the package wherein the name^a is contained, offered for sale or sold, a label upon the outside and face of which is distinctly and legibly printed in letters not less than one-half inch in length, the words "lard substitute," "adulterated lard" or "lard compound" or other appropriate words which shall correctly express its nature and use.

SEC. 41. *Possession of lard substitute evidence of intent to sell*. The having in possession of any lard substitute or adulterated lard compound, as hereinbefore defined, which is not branded or labeled as hereinbefore required or

^a So in Statutes.

directed upon the part of any manufacturer, trader or dealer, or any person engaged in the sale of such articles, shall, for the purpose of this Act, be deemed prima facie evidence of intent to sell or exchange the same.

Session Laws 1907-1908, ch. 37, pp. 418-419.

MEAT.

See General Food Laws, sec. 47, page 58.

PRESERVATIVES.

SEC. 42. *Preservatives prohibited; exemptions.* It shall be unlawful for any person to manufacture, sell or expose for sale or exchange any article of food to which has been added formaldehyde, borax, boracic acid, benzoic acid, sulphurous acid, salicylic^a acid, abradol^a beta-naphthal,^a flourine compounds^a saccharine, alcohol; provided that in the case of molasses and syrups and bleached dried fruits, that in the finished products sulphurous acid, flourine compound^a and chlorine are entirely removed subject to the rulings of the National Pure Food Commission. Provided, that the spreading of dry borax over the surface of meat cannot be construed to be a violation of this Act.

SEC. 50. *Preservatives added to oysters, etc.* Any corporation, firm or person, either in person or by an agent who shall sell or expose for sale within the state of Oklahoma any oysters, clams or other sea food products, to which salicylic^a acid, formaldehyde or any drug, or other preservative has been added or in preserving which any poisonous or deleterious substance has been used, shall be deemed guilty of a misdemeanor.

Session Laws 1907-1908, ch. 37, pp. 419-421.

SEA FOOD.

See Preservatives, sec. 50, above.

SPICES AND CONDIMENTS.

SEC. 46. *Compound spices must be so labeled; terms defined.* It shall be unlawful for any person to manufacture, sell, offer or expose for sale or exchange to the residents of this state, any spices and condiments,^a either ground or unground, which are adulterated with any foreign substance or substances within the meaning of this article, which are injurious to health and provided that when foreign substances are used, the package containing said article offered for sale shall contain the word "compound." The term spices and condiments as used herein shall^a embrace all substances known and recognized in commerce as spices, and used as condiments, whether the same be in natural state or in the form which would result from grinding, milling or mixing, or the compounding of the natural product.

Session Laws 1907-1908, ch. 37, p. 420.

VINEGAR.

See General Food Laws, sec. 55, page 58.

^a So in Statutes.

PORTO RICO.

GENERAL FOOD LAW.

SEC. 1. *Repeal.* Section 336 of the Penal Code is hereby repealed. [See U. S. Dept. Agr., Bureau of Chemistry, Bul. 69, Rev., pt. 7, p. 549.]

Approved March 12, 1908. Laws of 1908, p. 93.

474. *False statements of agents, etc.; false weight or measurement; penalties.* Every commission merchant, broker, agent, factor or consignee, who shall willfully and fraudulently make, or cause to be made, to the principal or consignor of such commission merchant, agent, broker, factor or consignee, a false statement concerning the price obtained for or the quality or quantity of any property consigned or intrusted to such commission merchant, agent, broker, factor or consignee, for sale, shall be deemed guilty of a misdemeanor and on conviction thereof shall be punished by fine not exceeding five hundred dollars, or imprisonment in jail not exceeding six months, or by both such fine and imprisonment. Every person who is putting up in any bale, bag, box, barrel or other package any sugar, tobacco, coffee, rice or other goods usually sold in bales, bags, boxes, barrels or other packages, by weight or otherwise, puts in or conceals therein any extraneous substance whatever for the purposes of fraudulently increasing the weight or measurement of such bale, bag, box, barrel or other package with intent thereby to sell the goods therein, or to enable another to sell the same, for more than the actual weight or measurement of such goods, is punishable by fine not less than twenty-five dollars for such offense, or confined in jail for not less than thirty days, or by both fine and imprisonment in the discretion of the court.—*As amended March 12, 1908; Laws of 1908, p. 93.*

Revised Statutes and Codes of 1902, Penal Code, ch. 8, p. 588.

MEAT.

SEC. 7. *Use of refrigerated meat in public institutions.* The Director of Health, Charities and Correction, is hereby authorized to determine whether refrigerated meat may or may not be supplied to institutions under his direction.

Approved March 12, 1908. Laws of 1908, p. 74.

RHODE ISLAND.

GENERAL FOOD LAWS.

SEC. 1. *Penalty for adulterating or misbranding; exemption of articles for export.* It shall be unlawful for any person, firm, or corporation to manufacture, sell, or offer for sale within this state, any drug or article of food which is adulterated or misbranded within the meaning of this act, and any person, firm, or corporation violating any of the provisions of this act shall be guilty of a misdemeanor, and shall, upon conviction, be punished for the first offense by a fine not exceeding fifty dollars, for the second offense by a fine not exceeding one hundred dollars, and for the third and each subsequent offense by a fine of two hundred dollars or imprisonment for one year: *Provided*, that no article shall be deemed misbranded or adulterated within the provisions of this act when intended for export to any foreign country and prepared or packed according to the specifications or directions of the foreign purchaser, when no substance is used in the preparation or packing thereof in conflict with the laws of the foreign country to which said article is intended to be shipped; but if said article shall be in fact sold or offered for sale for domestic use or consumption, then this proviso shall not exempt said article from the operation of any of the other provisions of this act.

[Secs. 2 and 3 pertain to drugs.]

SEC. 4. *Adulteration defined.* Food shall be deemed to be adulterated:

First.—If any substance has been mixed and packed with it so as to reduce or lower or injuriously affect its quality, strength, or purity. Second.—If any substance has been substituted wholly or in part for the article. Third.—If any valuable constituent of the article has been wholly or in part abstracted. Fourth.—If it is mixed, colored, powdered, coated, stained, or put up in a manner whereby damage or inferiority is concealed. Fifth.—If it contains any added poisonous or other added ingredient which may render such article injurious to health: *Provided*, that when in the preparation of food products for shipment they are preserved by any external application applied in such manner that the preservative is necessarily removed mechanically or by maceration in water, or otherwise, and directions for the removal of said preservative shall be printed on the covering of the package, the provisions of this act shall be construed as applying only when said products are ready for consumption. Sixth.—If it consists in whole or in part of a filthy, decomposed, or putrid animal or vegetable substance, or any portion of an animal unfit for food, whether manufactured or not, or if it is the product of a diseased animal, or one that has died otherwise than by slaughter.

SEC. 5. *Adulteration of confectionery defined.* Confectionery shall be deemed to be adulterated if it contains terra alba, barytes, talc, chrome yellow, or other mineral substances or poisonous colors or flavors, or other ingredients deleterious or detrimental to health, or any vinous, malt, or spirituous liquor or compound or narcotic drug.

SEC. 6. *Misbranding defined.* A drug or an article of food, or an article which enters into the composition of food, shall be deemed to be misbranded:

First.—If the package containing it, or the label on such package, shall bear any statement, design, or device regarding such article, or the ingredients or substances contained therein, which shall be false or misleading in any particular, or if the same is falsely branded as to the state, territory, or country in which it is manufactured or produced. Second.—If it be offered for sale as an imitation of, or under the name of, another article. Third.—If it is in the package form, and the contents are stated in the terms of weight or measure, the same is not plainly and correctly stated on the outside of the package. Fourth.—If the package contains a proprietary or patent medicine, or a proprietary or patent food, and the label fails to bear a statement of the quantity or the proportion of any alcohol, morphine, opium, cocaine, heroin, alpha or beta eucaine, chloroform, cannabis indica, chloral hydrate, or acetanilid or any derivative or preparation of any such substances contained therein: *Provided*, that the provisions of this section shall not apply to the sale and distribution of such proprietary or patent medicines or proprietary or patent foods as were in the possession of any dealer within this state at the time of the taking effect of this law.

SEC. 7. *Guaranty for protection of dealer.* No dealer shall be convicted under the provisions of this act, when he can establish a guaranty, signed by the wholesaler, jobber, manufacturer, or other party residing in the United States, from whom he purchases such articles, to the effect that the same is not adulterated or misbranded within the meaning of the food and drugs act of the United States, approved June 30, 1906, or of this act. Said guaranty, to afford protection, shall contain the name and address of the party or parties making the sale of such articles to such dealer, and in such case said party or parties shall be amenable to the prosecutions, fines, and other penalties which would attach, in due course, to the dealer under the provisions of this act.

SEC. 8. *Sampling; penalty for hindering execution of law.* Every person offering for or exposing for sale or delivering to a purchaser any drug or article of food included in the provisions of this act shall furnish to any commissioner, or other officer or agent appointed hereunder, who shall apply to him for the purpose and shall tender to him the value of the same, a sample or samples, of any drug or article of food which is in his possession, sufficient, after division into two equal or nearly equal parts, for the purpose of analysis. The official or agent thus taking said sample or samples shall then and there, in the presence of the person from whom he obtained it, unless said person refuse to witness the operation, divide said sample or samples into two equal or nearly equal parts or specimens, and seal and label the same, said label to state the kind of food or drugs, the date of such taking, and, if obtainable, the name of the person from whom they were taken; also, if obtainable, the name or names of the parties, if there be any, whom said person represents. Said official or agent shall then and there deliver one of said specimens to the person from whom the same were taken. If any such sample or samples so taken shall appear to be adulterated within the meaning of this act, notice in writing of the fact of such adulteration, containing a description of such sample or samples, shall forthwith be given by mail or otherwise, directed to the person from whom the same were obtained, to the address given by him at the time such sample or samples were taken, before any prosecution shall be instituted thereon: *Provided, however*, that if the person from whom such sample or samples are taken shall omit or refuse to give his name or address, such notice shall not be required. Whoever hinders, obstructs, or in any way interferes with any commissioner or other officer or agent appointed hereunder, in the performance of

his duty, shall, upon conviction, be fined a sum not exceeding one hundred dollars.

SEC. 9. *Seizure.* Any article of food or any drug that is adulterated or misbranded within the meaning of this act shall be liable to be proceeded against in the courts of this state within the county where found, and seized for forfeiture by the same process of law under which liquors illegally sold or for sale may be seized for forfeiture; and if such article or drug is condemned as being adulterated or misbranded or of a poisonous or deleterious character within the meaning of this act, it shall be disposed of by destruction or sale, as the court may direct, and the proceeds thereof, if sold, less the legal costs and charges, shall be paid into the treasury of the state: *Provided, however,* that upon the payment of the costs of such proceedings and the execution and delivery of a good and sufficient bond to the effect that such articles or drugs shall not be sold or otherwise disposed of contrary to the provisions of this act, the court may, by order, direct that such articles or drugs be delivered to the owner thereof. Either party may demand trial by jury of any issue of fact in any such case, and all such proceedings shall be at the suit of and in the name of the state.

SEC. 10. *Soaked canned goods.* All canned articles of food which have been prepared from dried products and have been soaked before canning shall be plainly marked by a brand or label having on its face the word "Soaked," in letters of legible type not smaller than eight-point (Brevier) caps.

SEC. 11. *Board of commissioners.* There shall be a board of food and drug commissioners, consisting of three members, who shall hold office for the term of their appointment, and until their successors, respectively, shall be elected and qualified to act.

At the January session of the general assembly in the year A. D. 1908, the governor, with the advice and consent of the senate, shall appoint three persons to be members of said board, one for a term ending January 31, 1910, one for a term ending January 31, 1912, and one for a term ending January 31, 1914.

At the January session of the general assembly in the year A. D. 1910, and in every second year thereafter, the governor, with the advice and consent of the senate, shall appoint a person to be a member of^a said board, and the person so appointed shall hold his office until the first day of February in the fifth year of his appointment. Any vacancy which may occur in said board when the senate is not in session shall be filled by the governor until the next session thereof, when he shall, with the advice and consent of the senate, appoint some person to fill such vacancy for the remainder of the term.

SEC. 12. *Duties of board; rules and standards; organization.* It shall be the duty of said board to enforce the provisions of this act. They shall adopt such rules, consistent with the provisions of this act, as may be necessary for its enforcement, and shall adopt rules regulating minimum standards of strength, purity, and quality for food and drugs, defining specific adulterations when such standards are not specified or fixed under this act or by the laws of this state, and subject to the provisions of this act, declaring the proper methods of collecting and examining drugs and articles of food; but such rules and standards shall not be more stringent than, nor conflict with, the rules and standards adopted, or which may hereafter be adopted, for the enforcement of the food and drug act of the United States, approved June 30, 1906, or of any food and drug act of the United States hereafter in force, regulating the misbranding or adulteration of food and drug products for interstate commerce: *Provided, however,* that in prosecutions under this act when the strength, quality, or purity of a drug or an article of food is in issue and the standard of strength,

^a So in Statutes.

quality, or purity of such drug or article of food is fixed by said board, proof that such drug or article of food is below the standard of strength, quality, or purity fixed by said board shall be evidence that such drug or article of food is adulterated within the meaning of this act.

The said commissioners shall have an office in the state house. They shall be allowed such office, traveling, and personal expenses as may be approved by the governor, to be paid, upon the order of the state auditor, out of any money in the treasury not otherwise appropriated.

They shall meet at least once in three months and as much oftener as may be necessary. They shall proceed to organize by the election of a chairman and an executive secretary, who shall be a practical chemist. Said board shall have authority to appoint such other agents as may be necessary to assist in the enforcement of this act. Said executive secretary and agents shall work under the direction of the said board of commissioners and shall perform such duties as the said board shall prescribe for them to perform.

SEC. 13. *Appropriation.* The sum of thirty-five hundred dollars is hereby appropriated annually, commencing January 1, 1909, from the treasury of the state, to be expended by the board of food and drugs commissioners, for the purpose of meeting the expenses incurred in the enforcement of this act, including fifteen hundred dollars the salary of an executive secretary, the cost of collection of samples, purchase of laboratory supplies, and aid in prosecuting offenders against this act.

SEC. 14. The sum of fifteen hundred dollars or as much thereof as may be necessary, including seven hundred and fifty dollars as recompense for the services of an executive secretary, is hereby appropriated out of the treasury of the state for the purpose of meeting the necessary expense of preparation and notification; and the state auditor is hereby directed to draw his order upon the general treasurer for the payment of the same upon the receipt of vouchers approved by the chairman and secretary of said board.

SEC. 15. *Milk, meat, feeding stuffs, and contagious disease laws not repealed.* This act shall not be construed to repeal Chapter 147 of the General Laws, entitled "Of milk," or any acts in amendment thereof or in addition thereto, or Chapter 131 of the General Laws, entitled "Of the inspection of beef and pork," or any acts in amendment thereof or in addition thereto, or an act entitled "An act authorizing the city of Providence to elect an inspector of beef and pork for said city," passed June 29, 1833, or sections 1 and 2 of Chapter 281 of the Public Laws, entitled "An act in amendment of and in addition to Title XIV, Chapter 74, of the Revised Statutes, 'Of regulations for the prevention of infectious and contagious diseases,'" passed March 5, 1858, or Chapter 631 of the Public Laws, entitled "An act regulating the sale of concentrated commercial feeding stuffs," passed at the January session, 1899.

SEC. 16. *Effect.* Sections 11, 12, and 14 of this act shall take effect immediately, and all other parts of this act shall take effect January 1, 1909.

Approved May 26, 1908. Public Laws passed at the January Session, 1908, ch. 1597, pp. 295-303.

CANNED GOODS.

See General Food Laws, page 71.

CONFECTIONERY.

See General Food Laws, page 69.

SOUTH CAROLINA.

RICE FLOUR.

SEC. 1. *Presence of chaff, etc., must be stated on label.* From and after the approval of this Act it shall be unlawful for any person to sell, or expose for sale, rice flour which contains chaff or any other adulteration, without giving notice by label or otherwise the nature and extent of such adulteration.

SEC. 2. *Adulterated rice flour liable to seizure.* Any rice flour so adulterated, which is sold or exposed to sale without being labeled or advertised as such, shall be liable to seizure and sale by any Magistrate having jurisdiction, on the prosecution of any person, the proceeds of such sale to be paid into the Treasury of the County in which such rice flour may be seized.

Approved February 17, 1908. Acts of 1908, No. 476, p. 1053.

VIRGINIA.

GENERAL FOOD LAWS.

SEC. 1. *Appointment of dairy and food commissioner.* Within thirty days after this act shall take effect, the governor, by and with the consent of the general assembly in joint session, shall appoint a suitable person to be dairy and food commissioner, which office is hereby created within the department of agriculture and immigration, and which commissioner so appointed shall hold his office until January thirty-one, nineteen hundred and twelve, and until his successor is appointed and qualified. At the regular session of the legislature in nineteen hundred and twelve, and every four years thereafter, the governor, by and with the advice and consent of the general assembly in joint session, shall appoint a dairy and food commissioner, who shall hold his office for the term of four years from the thirty-first day of January, in the year of his appointment, and until his successor is appointed and qualified.

SEC. 2. *Governor may remove commissioner.* The governor shall have the power to remove such commissioner any time, in his discretion, but the reasons for such removal shall be laid before the general assembly in joint session at the next regular or special session of the legislature thereafter; and in case of a vacancy in the office of commissioner from any cause, the governor shall appoint his successor to fill the unexpired term.

SEC. 3. *Oath and bond of commissioner.* Before entering upon the duties of his office, the person so appointed shall make, subscribe and file in the office of the secretary of the Commonwealth, the usual oath of office as provided for in the Constitution of this State, and shall enter into bond, payable to the Commonwealth, in the sum of five thousand dollars, with securities approved by the governor, conditioned for the faithful performance of his duties.

SEC. 4. *Salaries and assistants.* Said dairy and food commissioner shall receive an annual salary of two thousand five hundred dollars. There shall be a deputy dairy and food commissioner, who shall be appointed by the commissioner of agriculture and immigration and the dairy and food commissioner, acting jointly, subject to the confirmation of the State board of agriculture and immigration. The salary of the deputy commissioner shall be fifteen hundred dollars per annum. The said commissioners may also appoint by and with the advice of the board of agriculture and immigration such other special assistants as the proper performance of the duties of the office may require, which special assistants shall be paid for the time actually employed, as said commissioners and board may direct. The persons so appointed shall have power to administer oaths in all matters relative to the dairy and food laws, and shall take and subscribe to the constitutional oath of office, and file the same in the office of the secretary of the Commonwealth; and they shall hold office during the pleasure of the commissioners. The assistants shall have the same right of access to the places to be inspected as the said commissioner. The salaries and expenses authorized by this section shall be for the unexpired part of the fiscal year ending nineteen hundred and eight, and each fiscal year thereafter. Said salaries are to be paid monthly. The salaries and actual and necessary expenses of the said commissioner, deputy commissioner and

assistants, in the performance of their official duties, shall be audited by the State board of agriculture and immigration, and paid upon warrants issued by the dairy and food commissioner upon the State auditor. The board of agriculture and immigration shall provide office room and the necessary furniture and fixtures, and the necessary stationery, supplies and printing for the conduct of the business of said dairy and food commissioner, on his application to said board therefor. Said office shall be, and remain in the city of Richmond.

SEC. 5. *Chemical work.* The chemical work incident to the execution of the dairy and pure food laws shall be done in the chemical laboratory of the department of agriculture and immigration.

SEC. 6. *Duties of commissioner; analyses; inspection; penalty.* It shall be the duty of the dairy and food commissioner to carefully inquire into the dairy and food and drink products, and the several articles which are food or drinks, or the necessary constituents of the food or drinks, which are manufactured or sold, or exposed or offered for sale in this State, and he may, in a lawful manner, procure samples of the same, which shall be duly and carefully examined or analyzed by the State chemist, who shall report to the said commissioner the results of such examination or analyses; and it shall be the duty of the said commissioner to make a complaint against the manufacturer or vendor of any such food or drink or dairy products as are adulterated, impure or unwholesome, in contravention of the laws of this State, and furnish all evidence thereof to obtain a conviction of the offense charged. The dairy and food commissioner or his deputy, or any person appointed by him for that purpose, may make complaint and cause proceedings to be commenced against any person for enforcement of the laws relative to adulteration, impure or unwholesome food or drink, and in such cases he shall not be obliged to furnish security for costs, and shall have power, in the performance of his duties, to enter into any creamery, factory, store, salesroom, drug store, or laboratory, or place where he has reason to believe food and drink is made, stored, sold, or offered for sale, and open any cask, tub, jar, bottle or package containing, or supposed to contain, any article of food or drink, and examine or cause to be examined the contents thereof, and take therefrom samples for analysis. The person making such inspection shall take such samples of such article or product in the presence of at least one witness, and he shall, in the presence of said witness, mark or seal such sample, and shall tender at the time of taking to the manufacturer or vendor of such product, or to the person having the custody of the same, the value thereof, and the statement in writing for the taking of such sample. Whenever it is determined by the dairy and food commissioner, his deputy or assistants, that filthy or unsanitary conditions exist or are permitted to exist in the operation of any bakery, confectionery, or ice cream plant, or at any place where any food or drink products are manufactured, stored or deposited, or sold for any purpose whatever, the proprietor or proprietors, owner or owners of such bakery, confectionery or ice cream plant, or any person or persons owning or operating any plant where any food or drink products are manufactured, stored, deposited or sold, shall be first notified and warned by the said commissioner, his deputy or assistants, to place such bakery, confectionery, or ice cream plant, or any place where any food or drink products are manufactured, stored, deposited or sold, in a sanitary condition within a reasonable length of time; and any person or persons owning or operating any bakery, confectionery or ice cream plant, or any place where any food or drink products are manufactured, stored, deposited or sold, failing to obey such notice and warning, shall be guilty of a misdemeanor, and, upon conviction thereof, shall be punished by a fine of not less than twenty-five dollars nor more than three

hundred dollars and costs of prosecution, or imprisonment in the county or city jail not to exceed ninety days, or until such fine or costs are paid, or both fine and imprisonment, at the discretion of the court.

Sec. 7. *Seizures, sampling, and analysis; prosecution.* The dairy and food commissioner, his deputy, or any person by said commissioner duly appointed for that purpose, is authorized at all times to seize and take possession of any and all food and dairy products, substitutes therefor, or imitation thereof kept for sale, exposed for sale, or held in possession or under the control of any person which in the opinion of said commissioner, or his deputy, or such person by him duly appointed, shall be contrary to the provisions of this act or other laws which now exist or which may be hereafter enacted.

First. The person so making such seizure, as aforesaid, shall take from such goods as seized a sample for the purpose of analysis and shall cause the remainder to be boxed and sealed and shall leave the same in the possession of the person from whom they were seized, subject to such disposition as shall hereafter be made thereof according to the provisions of this act.

Second. The person so making such seizure shall forward the sample so taken to the dairy and food commissioner who shall turn over the same to the State chemist and the said chemist shall certify the results of such analysis, which certificate shall be prima facie evidence of the fact or facts therein certified to, in any court where the same may be offered in evidence.

Third. If upon such analysis it shall appear that said food or dairy products are adulterated, substituted, mis-branded, or imitated within the meaning of this act, said commissioner, or his deputy, or any person by him duly authorized may make complaint before any justice of the peace or police justice having jurisdiction in the city, village or magisterial district, where such goods were seized, and thereupon said justice of the peace shall issue his summons to the person from whom said goods were seized, directing him to appear not less than six or more than twelve days from the date of issuing of said summons and show cause why said goods should not be condemned and disposed of. If the said person from whom said goods were seized cannot be found, the said summons shall be served upon the person then in possession of the goods. The said summons shall be served at least six days before the time of appearance mentioned therein. If the person from whom said goods were seized cannot be found, and no one can be found in possession of said goods, and the defendants shall not appear on the return day, then said justice of the peace shall proceed in said cause in the same manner provided by law where a writ of attachment is returned not personally served upon any of the defendants and none of the defendants shall appear upon the return day.

Fourth. Unless cause to the contrary thereof is shown, or if said goods shall be found upon trial to be in violation of any of the provisions of this act or other laws which now exist or which may be hereafter enacted, it shall be the duty of said justice of the peace or police justice to render judgment that said seized property be forfeited to the State of Virginia, and that the said goods be destroyed or sold by the said commissioner for any purpose other than to be used for food. The mode of procedure before said justice shall be the same as near as may be in civil proceedings before justices of the peace. Either party may appeal to the circuit or corporation courts as appeals are taken from the justices' courts, but it shall not be necessary for the Commonwealth to give any appeal bond.

Fifth. The proceeds arising from any such sale shall be paid into the State treasury and credited to the general fund; provided, that if the owner or party claiming the property or goods so declared forfeited can produce and

prove a written guaranty of purity, signed by the wholesaler, jobber, manufacturer, or other party residing within this State from whom said articles were purchased, then the proceeds of the sale of such articles, over and above the costs of seizure, forfeiture and sale, shall be paid over to such owner or claimant to reimburse him, to the extent of such surplus, for his actual loss resulting from such seizure and forfeiture as shown by the invoice.

Sixth. It shall be the duty of the prosecuting attorney when called upon by said commissioner, or by any person by him authorized as aforesaid, to render any legal assistance in his power in proceeding under the provisions of this act, or any subsequent act relative to the adulteration of food, for the sale of impure or unwholesome food or food products.

SEC. 8. *Annual report; quarterly bulletin.* The dairy and food commissioner shall make an annual report to the commissioner of agriculture and immigration to be, by said commissioner of agriculture and immigration transmitted to the governor on or before the first day of January in each year, and which shall be printed and published on or before the first day of January next thereafter, which report shall cover the doings of his office for the preceding fiscal year, which shall show, among other things, the number of manufactories and other places inspected and by whom, the number of specimens^a of food articles analyzed and the State chemist's report upon each one; the number of complaints entered against persons for the violating of the laws relative to the adulteration of food, the number of convictions had, and the amount of fines imposed therefor, together with such recommendations relative to the statutes in force as his experience may justify. The dairy and food commissioner shall prepare, print and distribute to all papers of the State, and to such persons as may be interested or may apply therefor, a quarterly bulletin in suitable paper covers, containing results of inspections, the results of analyses made by the State chemist, with the popular explanation of the same, and such other information as may come to him in his official capacity relating to the adulteration of food and drink products and of dairy products, so far as he may deem the same of benefit and advantage to the public; also a brief summary of all the work done during the quarter by the commissioner and his assistants in the enforcement of the laws of the State, but not more than ten thousand copies of such quarterly bulletin shall be printed.

SEC. 9. *Penalty for hindering commissioner.* Any person who shall wilfully hinder or obstruct the dairy and food commissioner, or his deputy or other persons or assistants by him duly authorized, in the exercise of the powers conferred upon him by this act, shall be deemed guilty of a misdemeanor and on conviction shall be punished by a fine of not less than ten dollars nor more than one hundred dollars, or by imprisonment in the county or city jail for not less than ten days nor more than ninety days, or both such fine and imprisonment, in the discretion of the court.

SEC. 10. *Appropriation.* For the purpose of carrying out the provisions of this act the sum of seven thousand five hundred dollars is hereby appropriated for the fiscal year ending February twenty-eighth, nineteen hundred and nine, and in like manner for each fiscal year thereafter, there is hereby appropriated the sum of seven thousand five hundred dollars.

Approved March 11, 1908. Acts of 1908, ch. 188, p. 266.

Repeal. An act entitled an act to prevent the sale of adulterated and misbranded foods in the State of Virginia, approved February twenty-seventh,

^a So in Statutes.

nineteen hundred [Bul. 69 Rev., pt. 8, pp. 639-642], be and the same is, hereby repealed and be it further enacted by the general assembly of Virginia :

SEC. 1. *Sampling and analyses; appointments.* For the purpose of protecting the people of the State from imposition by the adulterating^a and misbranding of food, the dairy and food commissioner shall cause to be procured from time to time, and under the rules and regulations to be prescribed by him, with the approval of the board of agriculture and immigration in accordance with the provisions of this act, samples of food offered for sale in this State, and shall cause the same to be analyzed and examined microscopically or otherwise by the chemists or other experts of the department of agriculture and immigration; and he is hereby authorized to make such publication of the results of the examination, analyses, and so forth, as he may deem proper; and for the proper execution of the provisions of this act, the dairy and food commissioner shall with the approval of the board make such appointments as may be necessary and the board shall fix the compensation of such appointees.

SEC. 2. *Adulteration a misdemeanor; penalty.* No person, firm or corporation, either directly or through any agent, shall manufacture, sell, expose for sale or have in his possession with intent to sell, any article of food, which is adulterated or misbranded within the meaning of this act, and any person who shall violate any of the provisions of this act shall be guilty of a misdemeanor, and for such offense, shall be fined not exceeding two hundred dollars for the first offense, and for each subsequent offense not exceeding three hundred dollars, or be imprisoned not exceeding one year, or both, in the discretion of the court; and such fines less legal costs and charges, shall be paid into the treasury of the State.

SEC. 3. *Results of analysis as evidence; hearings.* The chemists or other experts of the department of agriculture and immigration shall make, by the methods in use at the time by the association of official agricultural chemists of the United States, examinations of specimens of food offered for sale in Virginia, which may be collected from time to time as prescribed by this act in various parts of the State; and if it shall appear from any such examinations that any such specimen is adulterated or misbranded within the meaning of this act, that notice thereof shall be given to the manufacturer, guarantor, or person from whom the sample was obtained. Any person so notified shall be given an opportunity to be heard under such rules and regulations as may be prescribed by the dairy and food commissioner and the commissioner and board of agriculture and immigration, and if it appears that any of the provisions of this act have been violated, the dairy and food commissioner shall certify the facts to the Commonwealth's attorney of the city or county in which the sample was obtained, and furnish the officer with a copy of the results of the analysis or other examinations of such article, duly authenticated by the analyst or other officer making such examination under the oath of such officer. In all prosecutions arising under this act the certificates of the analyst or other officer making the analysis or examination, when duly sworn to by such officer, shall be prima facie evidence of the fact or facts therein certified.

SEC. 4. *Prosecution.* It shall be the duty of every Commonwealth's attorney to whom the dairy and food commissioner shall report any violation of this act to cause the proceedings to be commenced and prosecuted without delay for the fines and penalties in such cases prescribed.

SEC. 5. *"Food" defined.* The term "food" as used in this act shall include all articles used for food, drink, confectionery, or condiment by man or other animals, whether simple, mixed, or compound.

^a So in Statutes.

SEC. 6. *Adulteration defined; confectionery; food.* For the purpose of this act an article shall be deemed to be adulterated:

In the case of confectionery:

First. If it contains terra alba, barytes, talc, chrome yellow, or other mineral substance or poisonous color or flavor, or other ingredient deleterious or detrimental to health, or any vinous, malt, or spirituous liquor or compound or narcotic drug.

In case of other food:

First. If any substance has been mixed or packed with it, so as to reduce or lower or injuriously affect its quality or strength.

Second. If any substance has been substituted wholly or in part for the article.

Third. If any valuable constituent of the article has been wholly or in part abstracted.

Fourth. If it be mixed, colored, powdered, coated, polished or stained in a manner whereby damage or inferiority is concealed.

Fifth. If it contains any added poisonous or other added deleterious ingredient which may render such article injurious to health. Provided, that when in the preparation of food products for shipments they are preserved by any external application in such manner that the preservative is necessarily removed mechanically, or by maceration in water, or otherwise, and directions for the removal of said preservative shall be printed on the covering of the package or furnished with the article, the provisions of this act shall be construed as applying only when said products are ready for consumption.

Sixth. If it consists in whole or in part of diseased, filthy, decomposed, or putrid animal or vegetable matter, or any portion of an animal unfit for food whether manufactured or not, or if it is the product of a diseased animal, or one that had died otherwise than by slaughter.

Seventh. If the containing vessel or any part of it be of such composition as will be acted upon, in the ordinary course of use, by the contents thereof in such a way as to produce an injurious, deleterious, or poisonous compound.

SEC. 7. *Misbranding defined.* The term "misbranded" as used herein shall apply to all articles of food, or articles which enter into the composition of food, the package or label of which shall bear any statement, design or device regarding such article, or the ingredients or substance contained therein, which shall be false or misleading in any particular, and to any food product which is falsely branded as to the State, territory, or country in which it is manufactured or produced.

For the purpose of this act an article shall also be deemed misbranded:

First. If it be an imitation of, or offered for sale under the distinctive name of another article.

Second. If it be labeled or banded^a so as to deceive or mislead the purchaser, or purport to be a foreign product when not so, or if the contents of the package as originally put up shall have been removed in whole or part, and other contents shall have been placed in such package, or if it fail to bear a statement on the label of the quantity or proportion of any morphine, opium, cocaine, heroin, alpha or beta eucaine, chloroform, cannabis indica, chloral hydrate, or acetanilide or any derivative or preparation of any such substance contained therein.

Third. If in package form, and the contents are stated in terms of weight or measure, they are not plainly and correctly stated on the outside of the package.

Fourth. If the package or its label shall bear any statement, design, or device regarding the ingredients or substance contained therein, which statement,

^a So in Statutes.

design, or device shall be false or misleading in any particular: Provided, that an article of food which does not contain any added poisonous or deleterious ingredients shall not be deemed to be adulterated or misbranded in the following cases:

First. In the case of mixtures or compounds which may be now or from time to time hereafter known as articles of food under their own distinctive names, and not an imitation of, or offered for sale under the distinctive name of, another article of food, if the name be accompanied on the same label or brand with a statement of the place where said article has been manufactured or produced.

Second. In the case of articles labeled, branded, or tagged so as to plainly indicate that they are compounds, imitations or blends, and having the word "compound," "imitation," or "blend" as the case may be, plainly stated on the package in which such article is offered for sale: provided, the labeling is according to the rules prescribed by the dairy and food commissioner with the approval of the commissioner and board of agriculture and immigration:

Provided, that the term "blend" as used herein shall be construed to mean a mixture of like substances, not excluding harmless coloring or flavoring ingredients used for the purpose of coloring and flavoring only: and provided further that nothing in this act shall be construed as requiring or compelling proprietors or manufacturers of proprietary foods which contain no unwholesome added ingredient to disclose their trade formulas, except in so far as the provisions of this act may require to secure freedom from adulteration and misbranding.

SEC. 8. *Sanitary conditions for handling of human food, especially meats.* It shall be unlawful for any person or persons, firm or corporation, to sell, or to have in his possession with intent to sell for human food, meat or meat food products which has been slaughtered, prepared, or kept where the sanitary conditions, are such that the meat or meat food products are rendered unhealthy, unwholesome, or otherwise unfit for human food.

All peace and health officers shall have the power and are required to seize any animal carcass or parts of carcasses which are intended for sale or offered for sale for human food, which have been slaughtered and prepared, handled or kept under unsanitary conditions, and shall deliver the same forthwith to and before the nearest police judge or justice of the peace, together with all information obtained, and said police judge or said justice of the peace shall, upon sworn complaint being filed, issue warrant for the arrest of all persons who have violated the provisions of this section, and proceed to try the case. Any person, persons, firm or corporation found guilty of violating the provisions of this section shall be fined not less than ten nor more than one hundred dollars, and the meat in question shall be destroyed.

SEC. 9. *Guaranty.* No dealer shall be prosecuted under the provisions of this act when he can establish a guaranty signed by a wholesale dealer, manufacturer or other party, residing in Virginia, from whom he purchased such articles, to the effect that the same is not adulterated or misbranded within the meaning of this act, designating it. Provided, however, that if the article in question is in a broken or open package, said guaranty shall not afford immunity from prosecution, unless such dealer shall furnish satisfactory proof that the article has not been changed in quality. The affidavit of such person shall be accepted as such proof, and the person making such affidavit falsely shall be guilty of perjury, and punished accordingly: Said guaranty, to afford protection, shall contain the name and address of the party or parties making the

sale of such articles to such dealer, and in such cases said party or parties shall be amenable to to^a the prosecutions, fines, and other penalties which would attach in due course, to the dealer under the provisions of this act: provided, that the above guaranty shall not afford protection to any dealer after the first offense in connection with a product from a particular wholesale dealer or manufacturer.

SEC. 10. *Standards.* The dairy and food commissioner with the approval of the commissioner and board of agriculture and immigration shall from time to time, fix and publish standards or limits of variability permissible in any article of food and these standards when so published shall be the standards before all courts: provided, that when standards have been or may be fixed by the secretary of agriculture of the United States, they shall be accepted by the department of agriculture and immigration and published as standards for Virginia, but said standards shall not go into effect until a reasonable time after publication. The dairy and food commissioner, with the approval of the commissioner and board of agriculture and immigration shall have authority to make uniform rules and regulations for carrying out the provisions of this act.

SEC. 11. *Sampling.* Every person who exposes or offers for sale or delivers to a purchaser any food, shall furnish within business hours and upon tender and full payment of the selling price, a sample of such food, to any person duly authorized to secure the same, and who shall apply to such manufacturer or vendor or person delivering such food to a purchaser for such sample in sufficient quantity for the analysis of such article or articles in his possession. Samples may be purchased on the open market and shall be representative samples; the collector shall also note the name of the vendor and agent through whom the sale was actually made, together with date of purchase, and all samples not taken in unbroken and sealed original packages shall be sealed by the collector in the presence of the vendor with a seal provided for the purpose.

When possible, samples shall be unbroken and sealed original packages, or taken out of unbroken and sealed original packages. Three like samples shall be obtained where the article is in the original package, or, if not in the original package, the sample obtained shall be divided into three equal parts and each part shall be labeled with the marks, brands or tags upon the package, carton, container, wrapper or accompanying printed or written matter. One sample shall be delivered to the party from whom purchased, or to the party guaranteeing such merchandise; two samples shall be sent to the dairy and food commissioner, one of which is to be analyzed, as provided in this act and the other shall be held under seal by the dairy and food commissioner.

SEC. 12. *Penalty for hindering enforcement.* Any manufacturer, dealer or person who refuses to comply upon demand with the requirements of this act or who shall impede, obstruct, hinder or otherwise prevent or attempt to prevent any chemist inspector or other person in the performance of his duty in connection with this act, shall be guilty of a misdemeanor, and upon conviction be fined not less than ten dollars nor more than one hundred dollars, or be imprisoned not more than one hundred days, or both, in the discretion of the court; and said fines, less the legal costs, shall be paid into the treasury of the State.

SEC. 13. *"Person" defined.* The word "person" as used in this act shall be construed to import both the plural and the singular, as the case demands, and shall include partnership, corporations, companies, societies and associations. When construing and enforcing the provisions of this act, the act, omission or

^a So in Statutes.

failure of any officer, agent or other individual acting for or employed by any partnership, corporation, company, society, or association within the scope of his employment or office, shall in every case be, also deemed the act, omission, or failure of such partnership, corporation, company, society, or association, as well as that of the individual.

SEC. 14. *Seizure and condemnation.* Any person, firm, or corporation who shall manufacture, sell or offer for sale any article of food that is adulterated within the meaning of this act, shall be guilty of a misdemeanor, and in addition to being subject to the penalties already provided in this act, the article of food shall be subject to seizure and condemnation, followed by sale or destruction.

Approved March 14, 1908. Acts of 1908, ch. 372, pp. 654-659.

CONFECTIONERY.

See General Food Laws, sec. 6, page 79.

DAIRY PRODUCTS.

SEC. 11. *Investigation of creameries, cheese and milk factories, etc.; assistants.* It shall be the duty of the dairy and food commissioner to foster and encourage the dairy industry of the State, and, for that purpose he shall investigate the general conditions of the creameries, cheese factories, condensed milk factories, skimming stations, milk stations and farm dairies in this State, with full power to enter upon any premises for such investigation, with the object in view of improving the quality and creating and maintaining uniformity of the dairy products of the State; and should it become necessary in the judgment of the dairy and food commissioner, he may cause instruction to be given in any creamery, cheese factory, condensed milk factory, skimming station, milk station or farm dairy, or in any locality of this State, and in order to secure the proper feeding and care of cows, or the practical operation of any plant producing dairy products, and in order to procure such a uniform and standard quality of dairy products in this State, he shall furnish a sufficient number of competent assistants, the appointment of whom is provided for in section four of this act, and they shall be duly qualified to act as such assistants.

SEC. 12. *Penalty for furnishing unclean milk to factories.* Whenever it is determined by the dairy and food commissioner, his deputy or assistants, that any person is using, selling or furnishing to any skimming station, creamery, cheese factory, condensed milk factory, milk depot, farm dairy, milk dealer, the retail trade or to any consumer of milk, any impure or unwholesome milk or cream, which impurity or unwholesomeness is caused by the unsanitary or filthy conditions of the premises where cows are kept or by the unsanitary or filthy care or handling of the cows, or from the use of unclean utensils or from unwholesome food, or from any other cause, the person so using, selling or furnishing to any skimming station, creamery, cheese factory, condensed milk factory, milk depot, farm dairy, milk dealer, the retail trade or to any consumer of milk, any such milk or cream, shall first be notified and warned by the said commissioner, his deputy or assistants not to use, sell or furnish such milk or cream to such skimming station, creamery, cheese factory, condensed milk factory, milk depot, farm dairy, milk dealers, the retail trade or to any consumer of milk, and any person failing to obey such notice and warning and continuing to use, sell or furnish to any skimming station, creamery, cheese

factory, condensed milk factory, farm dairy, milk dealer or to the retail trade such impure or unwholesome milk or cream, shall be guilty of a misdemeanor, and, upon conviction thereof shall be punished by a fine not less than ten dollars nor more than fifty dollars and costs of prosecution or imprisonment in the county or city jail not to exceed ninety days or until such fine and costs are paid or both fine and imprisonment at the discretion of the court.

SEC. 13. *Penalty for unsanitary conditions of creameries, etc.* Whenever it is determined by the dairy and food commissioner, his deputy or assistants, that unsanitary conditions exist, or are permitted to exist, in the operation of any skimming station, creamery, cheese factory, condensed milk factory, milk depot, or farm dairy, the proprietor or proprietors or manager of said skimming station, creamery, cheese factory, condensed milk factory, milk depot, or farm dairy, shall be first notified and warned by the said commissioner, his deputy or assistants, to place such skimming station in a sanitary condition within a reasonable length of time; and any person or persons owning or operating such skimming station, creamery, cheese factory, condensed milk factory, milk depot, or farm dairy, failing to obey such notices and warnings, shall be guilty of a misdemeanor, and upon conviction thereof, shall be punished by a fine of not less than twenty-five dollars nor more than three hundred dollars, and cost of prosecution, or imprisonment in the county jail not to exceed ninety days, or until such fine and costs are paid, or both fine and imprisonment, at the discretion of the court.

SEC. 14. *Registration of creameries, cheese factories, etc.* It shall be the duty of the proprietor or proprietors of every skimming station, creamery, cheese factory, condensed milk station, or milk depot, in the State where milk or cream is received, by purchase or otherwise, from three or more persons, to register with the dairy and food commissioner, on or before April first of each year, upon blanks furnished by said official, the location of such skimming station, creamery, cheese factory, condensed milk factory, or milk depot, and the name of its owner or owners and manager. And it shall be the duty of the proprietor or proprietors of every skimming station, creamery, cheese factory, condensed milk factory or milk depot, in this State, where milk or cream is received, by purchase or otherwise, from three or more persons, to file a report with the dairy and food commissioner, said report to be made on or before April first of each year, upon blanks furnished by said official, and to show the amount of milk or cream received by said skimming station, creamery, cheese factory, condensed milk factory, or milk depot during the year ending December thirty-first preceding; and said report shall show the amount of butter, cheese, or condensed milk, manufactured during the year, together with a list of the names and post-office addresses of the patrons of said skimming station, creamery, cheese factory, condensed milk factory,² or milk depot. Every skimming station, creamery, cheese factory, condensed milk factory, or milk depot, so registering and so reporting, shall pay to the office of the State dairy and food commissioner an annual registration fee of five dollars, to be paid at the time of such registration. The money so collected by the dairy and food commissioner shall be paid into the State treasury, and be used to help defray the expenses of the office of the dairy and food commissioner in addition to the annual appropriation therefor.

SEC. 15. [Relates to commercial feeding stuffs.]

SEC. 16. *Annual report of commissioner.* The published annual report of the dairy and food commissioner, which shall be made to the commissioner of agriculture and immigration, shall include a complete accounting of all moneys received and expended by the said commissioner for the period covered by said report.

SEC. 17. *Enforcement of food laws by dairy and food commissioner.* The enforcement of all existing laws to prevent the manufacture and sale of adulterated and misbranded articles of food heretofore placed under the direction of the commissioner and the board of agriculture and immigration, shall hereafter be placed under the dairy and food commissioner, and shall be enforced by him and under his direction; and all books, papers, and matters referring to the enforcement of such laws shall be transferred to the office of the dairy and food commissioner.

SEC. 18. *Effect.* An emergency existing, because of the large and unlawful sale of adulterated and misbranded food products, this act shall take effect from its passage.

Approved March 11, 1908. Acts of 1908, ch. 188, pp. 266-274.

MEATS.

See General Food Laws, sec. 8, page 80.

WISCONSIN.

General food and dairy laws and laws regulating the sale of bread, meat, and sirup, as amended July 9, 1907, are given in Bulletin 112, Part II, page 152, having been included for convenience in that compilation, which covered only laws for the year ending June 30, 1907.

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U. S. DEPARTMENT OF AGRICULTURE
BUREAU OF CHEMISTRY

SEP 17 1909

Issued June 7, 1909.

U. S. DEPARTMENT OF AGRICULTURE.

BUREAU OF CHEMISTRY—BULLETIN No. 122.

H. W. WILEY, Chief of Bureau.

PROCEEDINGS
OF THE
TWENTY-FIFTH ANNUAL CONVENTION
OF THE
ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS,
HELD AT
WASHINGTON, D. C., NOVEMBER 12-16, 1908.

EDITED BY
HARVEY W. WILEY,
SECRETARY OF THE ASSOCIATION.



WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1909.

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1909.

LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., January 15, 1909.

SIR: I have the honor to submit for your approval the Proceedings of the Twenty-fifth Annual Convention of the Association of Official Agricultural Chemists. The reports have been prepared in the most concise form practicable in consideration of the detailed and technical character of the work, all general discussion being practically eliminated. I recommend that these proceedings be published as Bulletin 122 of the Bureau of Chemistry.

Respectfully,

H. W. WILEY,
Chief of Bureau.

HON. JAMES WILSON,
Secretary of Agriculture.

(2)

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PROCEEDINGS OF THE TWENTY-FIFTH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

FIRST DAY.

THURSDAY—MORNING SESSION.

The twenty-fifth annual convention of the Association of Official Agricultural Chemists was called to order by the president, Mr. Harry Snyder, of St. Anthony Park, Minnesota, on the morning of November 12, in the Annex Hall of the Normandie Hotel, Washington, D. C.

Two hundred and sixteen members and visitors registered during the convention, constituting the largest attendance ever recorded. The list is as follows:

MEMBERS AND VISITORS PRESENT.

Adams, Arthur B., Bureau of Internal Revenue, Washington, D. C.
Albrech, Maximilian C., U. S. Food and Drug Inspection Laboratory, Pittsburg, Pa.
Allen, Robert McD., Agricultural Experiment Station, Lexington, Ky.
Alwood, William Bradford, "Stonehenge" Laboratories, Charlottesville, Va.
Amoss, Harold L., Bureau of Chemistry, Washington, D. C.
Averitt, S. D., Agricultural Experiment Station, Lexington, Ky.

Bailey, Herbert S., Bureau of Chemistry, Washington, D. C.
Baker, E. L., Geneva, N. Y.
Balcom, R. Wilfred, Food and Drug Inspection Laboratory, New York, N. Y.
Farber, Kate G., Bureau of Chemistry, Washington, D. C.
Barnard, Harry E., State Food and Dairy Commission, Indianapolis, Ind.
Bartlett, James M., Agricultural Experiment Station, Orono, Me.
Bates, Carleton, Bureau of Chemistry, Washington, D. C.
Beal, W. H., Office of Experiment Stations, Washington, D. C.
Bell, James Munsie, Bureau of Soils, Washington, D. C.
Bidwell, George L., Bureau of Chemistry, Washington, D. C.
Bigelow, Willard D., Bureau of Chemistry, Washington, D. C.
Billings, George A., Department of Agriculture, Washington, D. C.
Bowker, W. H., Bowker Fertilizer Company, Boston, Mass.
Boyle, Martin, Bureau of Chemistry, Washington, D. C.
Boyles, Frank M., Bureau of Chemistry, Washington, D. C.
Breazeale, James Frank, Bureau of Chemistry, Washington, D. C.
Breckenridge, John E., American Agricultural Chemical Company, New York, N. Y.

Bridges, Benjamin H., Food and Drug Analyst to State of Florida, Tallahassee, Fla.
 Brinton, Clement S., U. S. Food and Drug Inspection Laboratory, Philadelphia, Pa.
 Broughton, Levin B., Agricultural Experiment Station, College Park, Md.
 Browne, Charles A., New York Sugar Trade Laboratory, New York, N. Y.
 Bryan, A. Hugh, Bureau of Chemistry, Washington, D. C.
 Bryan, Thomas J., State Analyst, Chicago, Ill.
 Burnet, Wallace C., U. S. Food and Drug Inspection Laboratory, Savannah, Ga.
 Burnett, Lyle B., Bureau of Chemistry, Washington, D. C.

Campbell, Walter Gilbert, Bureau of Chemistry, Washington, D. C.
 Carpenter, Frank B., Virginia-Carolina Chemical Company, Richmond, Va.
 Carroll, John S., German Kali Works, Atlanta, Ga.
 Castleman, Philip, Department of Agriculture, Washington, D. C.
 Cathcart, Charles S., Agricultural Experiment Station, New Brunswick, N. J.
 Cavanaugh, George W., State College of Agriculture, Cornell University, Ithaca, N. Y.
 Chace, E. M., Bureau of Chemistry, Washington, D. C.
 Chapin, Robert M., Bureau of Animal Industry, Washington, D. C.
 Chesnut, Victor King, Bureau of Chemistry, Washington, D. C.
 Church, C. G., Bureau of Chemistry, Washington, D. C.
 Cochran, C. B., Department of Agriculture, West Chester, Pa.
 Cole, Frank, College Park, Md.
 Collins, Arthur T., Philadelphia, Pa.
 Collins, Paul, Agricultural College, College Park, Md.
 Collins, William Dennis, Bureau of Chemistry, Washington, D. C.
 Cook, Frank C., Bureau of Chemistry, Washington, D. C.

Davidson, Robert James, Polytechnic Institute, Washington, D. C.
 Deemer, Ralph B., College Park, Md.
 Denis, Willey, Bureau of Chemistry, Washington, D. C.
 Dietrich, Harry W., Noble'sville, Ind.
 Dodge, C. O., Bureau of Chemistry, Washington, D. C.
 Donk, M. G., Bureau of Chemistry, Washington, D. C.
 Doolittle, Roscoe E., U. S. Food and Drug Inspection Laboratory, New York, N. Y.
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 Edmond, Herman D., Agricultural Experiment Station, Storrs, Conn.
 Ellett, Walter B., Agricultural Experiment Station, Blacksburg, Va.
 Emery, James Armitage, Department of Agriculture, Washington, D. C.
 Emslie, Benjamin Leslie, Toronto, Canada.

Feldstein, Leonard, Department of Agriculture, Washington, D. C.
 Fetzer, Lewis William, College Park, Md.
 Fischer, Louis A., Bureau of Standards, Washington, D. C.
 Forst, Leo B., Bureau of Internal Revenue, Washington, D. C.
 Fox, Paul J., Bureau of Chemistry, Washington, D. C.
 Frear, Julia Reno, State College, Pa.
 Frear, William, Agricultural Experiment Station, State College, Pa.
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 Fuller, F. D., Department of Agriculture, Harrisburg, Pa.
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- Valaer, Peter, jr., Bureau of Internal Revenue, Washington, D. C.
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 Withers, W. A., Agricultural Experiment Station, Raleigh, N. C.
- Young, William J., Bureau of Chemistry, Washington, D. C.

REPORT ON FOOD ADULTERATION.

By H. F. BARNARD, *Referee*.

The demand for analytical data bearing on the composition of normal and abnormal food products that has arisen in the past three years because of the enactment of food legislation has shown most clearly the absolute necessity for accurate, precise, and at the same time rapid methods for food analysis. It did not at first appear that the

problems of the food analyst were greatly different from those of the analytical chemist, but as the work has developed, a new literature and a new chemistry have by rapid evolution been added to the broad field of chemical science. The boast of the manufacturing chemist that he is always a year in advance of the official chemist who is hunting down iniquities is a constant stimulus and makes necessary the continual development of new methods of analysis and the refinement of old practices.

No radical departure from established methods is advocated by those who have studied the different phases of food adulteration this past year, but the reports of the associate referees show the necessity for continued research.

On fruit products, baking powder and baking chemicals, fats and oils, condiments other than spices, and the determination of water in foods no reports were made. These subjects are all worthy of careful study, and it is to be hoped that this coming year they may be taken up.

I take occasion to call the attention of all food chemists to the imperative necessity for the adoption of uniform methods of analysis which have been proved accurate and reliable. The work of this association is most valuable in providing official methods, but no association can compel chemists to employ standard methods or insist upon more careful analytical work. The food analyst is to-day working constantly in the limelight and his results very frequently are carried to the courts and are subject to the scrutiny of expert chemists and the counsel for the defendant. In too many cases it has appeared that the results of analyses have been published and even used in court which later were found to be inaccurate, thus compelling those responsible for the publication and use of such reports to make public retraction. The value of our work is greatly impaired by the constant recurrence of such mistakes. The necessity for more careful work is well shown by data published in the Proceedings for last year, where chemists analyzing similar condensed milks report an ash content varying from 1.34 to 2.17 per cent and a fat content varying from 7.50 to 9.24 per cent. If the fat in the original milk is determined in these samples on the ash basis, in one case the original milk content is 4.2 per cent fat, in the other case 2.56 per cent, figures which indicate that the same sample of evaporated milk was in one instance made from whole milk and in the other from skimmed milk.

Attention is again called to the fact that we have no satisfactory alcohol table which is accepted by all chemists as a standard. The several alcohol tables now in use, namely, those published in the Official and Provisional Methods of Analysis, the tables given in the United States Pharmacopœia, and those in use by the Internal Revenue Bureau, are not alike. More than that, they are all calculated at 60° F., instead of at the generally accepted standard temperature of 20° C. Can not this association be of assistance to the puzzled chemist who is constantly compelled to recalculate and correct his results and who is confronted in court by alcohol percentages so different from his as to discredit his testimony, but which when calculated to the same basis on the same table are found to be identical?

The Bureau of Standards may well cooperate with the committee from this association for the purpose of revising the alcoholometric tables, and it is recommended that a committee be appointed for this purpose.

REPORT ON WINE.

By JULIUS HORTVET, *Associate Referee.*

OUTLINE OF THE WORK.

On February 28, after some preliminary correspondence, the referee on wine sent out the following letter, accompanied by methods of analysis for alcohol, extract, glycerol, ash, fluorids, and total sulphurous acid, substantially as given in Bulletin 107 and in Windisch's *Untersuchung des Weines*. On June 11 these instructions

were supplemented by a further letter submitting a modified method for the determination of volatile and fixed acids. The subjects reported upon included the determination of glycerol in wines, the examination of natural coloring matter in wines, and the determination of total, fixed, and volatile acids; besides which there was submitted a special report on the determination of reducing sugars, by R. M. West. These papers, together with the letter of instructions and a statement of the modified methods which were studied, constitute the report of referee.

INSTRUCTIONS.

FEBRUARY 28, 1908.

DEAR SIR: I send you herewith an outline of methods for analysis of wine. It is my desire that you subject these methods to careful investigation, using for the purpose samples of your own collection. In general the plan of work for this year will be to allow each collaborator considerable latitude as to how he is to conduct his work. In other words, you are requested to make an independent investigation of all or as many of these methods as possible and prepare a paper giving your results and criticisms. Special attention is directed to the following points:

(1) The change to 20° C. as the standard temperature for specific gravity and alcohol determinations.

(2) Uniformity of terms in which to express results, considering especially (a) the idea of expressing all results when possible in grams per 100 cc of sample, (b) the idea of expressing total, volatile, and fixed acids as cubic centimeters of normal acid in 100 cc of sample.

(3) Improvements in the method for determining glycerol.

(4) A thorough trial of the new method of determining volatile and fixed acids (see below). A drawing and a description of the apparatus used are given in this connection (see page 21).

(5) A criticism of the uranium method for determining phosphoric acid * * *.

(6) A thorough trial of the scheme for examination of the natural coloring matter of wines, using for the purpose samples of the red-wine class.

(7) A criticism of the method for detecting fluorids and the method for determining sulphurous acid.

(8) An investigation and criticism of any of the other provisional methods for wines. Consider especially the volumetric method for determining reducing sugars.

It is desired that the result of your investigations be reported in full, showing all important numerical data, conclusions, and recommendations. * * *

PROPOSED METHODS FOR THE DETERMINATION OF TOTAL, VOLATILE, AND FIXED ACIDS.

Total acids.^a

Measure 10 cc of the sample into a 300 cc flask, add 100 to 200 cc of recently boiled distilled water, according to the color of the wine, and boil three minutes under a reflux condenser. After cooling add 2 or 3 drops of phenolphthalein and titrate with tenth-normal sodium hydroxid. Express the result for total acids as cubic centimeters of normal acid in 100 cc of the wine.

Volatile and fixed acids.

The apparatus to be used consists of a cylindrical flat bottomed flask of about 300 cc capacity, provided with an elongated wide neck. Into the neck of this flask is fitted by means of a short section of thick rubber tubing a cylindrical shaped flask in the bottom of which is a small opening leading inward through a siphon-shaped tube bent back upon itself and terminating at a point close to the bottom. The inner flask is connected to a condenser by means of a bent tube and safety bulb. In the stopper is also fitted a small funnel provided with a glass stop-cock. The distillate from the condenser is received in a cylindrical graduate.

Pour 100 cc of recently boiled distilled water into the larger flask, tightly fit the smaller flask into the wide neck, run in 10 cc of wine through the funnel, rinse out the funnel with a little water, close the stop-cock and heat the water to boiling. The steam passing through the siphon tube and through the wine carries out the volatile acids. When 50 cc of distillate have passed over, empty the graduate and continue the

^a Pierre Breteau, Guide pratique des falsifications et alterations des substances alimentaires, p. 318.

distillation. Titrate the 50 cc distillate with one-tenth normal sodium hydroxid, using phenolphthalein as an indicator.

Stop the distillation when an additional 10 cc of distillate requires only one drop of the standard alkali solution to neutralize. Usually 80 cc of distillate will include practically all of the volatile acids.

On cooling the apparatus the liquid remaining in the inner flask is siphoned into the outer flask. Rinse out the remaining small amount of sample by running several portions of hot water through the funnel tube and disconnect the two flasks. In case of a light colored wine or a white wine, add 100 to 200 cc of recently boiled distilled water and titrate with one-tenth normal sodium hydroxid, using phenolphthalein as an indicator. In the case of a highly colored wine, after cooling the liquid make up to 100 cc, measure out 25 cc, dilute with recently boiled distilled water and titrate as before. Express the results for volatile and fixed acids as cubic centimeters of normal acid in 100 cc of the wine.

THE DETERMINATION OF GLYCEROL IN WINES.

The method submitted to collaborators has been subjected to trial on a dozen samples of genuine California wines. After the residues obtained by the method had been weighed, they were analyzed for glycerol by the acid-dichromate oxidation method, as follows:

The residue was dissolved in a little distilled water, filtered and washed through a previously dried and weighed small filter, and the solution made up with water to 50 or 100 cc, the volume depending on the amount of the dissolved residue. An aliquot portion of the solution (equivalent to from 0.3 to 0.5 gram of residue) was run into a 200 cc beaker, 20 cc of sulphuric acid (1 : 1) and 50 cc of standard potassium dichromate solution (1 cc equivalent to 0.01 gram of glycerol) run in and the beaker placed in boiling water. During the heating the strength of a prepared solution of ferrous-ammonium sulphate (240 grams in 1,000 cc) was determined by titration with the dichromate. At the end of two hours the beaker was removed from the boiling water, 100 cc of water added, and the excess of dichromate titrated with the ferrous-ammonium sulphate. From the result of the titration the weight of the oxidized glycerol was calculated.

A sample of chemically pure glycerol, which by specific gravity determination and refractometer reading was shown to be 99.3 per cent pure, gave by this method 97.7 per cent pure glycerol by weight. The filter containing the residue insoluble in water was again dried at 100° C., cooled in a desiccator and weighed. Tannin was determined in another aliquot portion of the solution by the official provisional method given for tannin in wine. The results of these determinations are shown in the accompanying table. In three instances, owing to insufficient material, no results for tannin were obtained.

There seem to be no means of estimating the loss of glycerol which takes place during the determination. It appears, however, that the material which is extracted and weighed as glycerol is never pure glycerol, as is generally assumed. The proportion of glycerol obtained by the method of oxidation ranges from 84.7 to 88.5 per cent of the weighed residues. Also it appears that in some cases a considerable amount of the residue consists of matter insoluble in water, and also tannin.

Mr. C. S. Ash, chemist of the California Wine Association, recognizes the need of an improved method for the determination of glycerol, and describes the following, which has been employed in his laboratory:

Measure out 100 cc of wine in a porcelain dish, evaporate to a thick sirup, then make alkaline with milk of lime and evaporate almost to dryness. Dissolve out the glycerol with successive portions of boiling hot alcohol, evaporate the alcohol to about 5 cc, transfer to a stoppered flask or cylinder of 100 cc capacity, make up to 100 cc with ætic ether, and allow to stand over night. Filter the liquid from the precipitate, evaporate off, and dry the glycerol to constant weight at a temperature not above 55° or 60° C.

The method is said to give fairly uniform results, and the most unsatisfactory part of the procedure is the evaporation of the solvent. A small percentage of glycerol is undoubtedly lost, especially toward the end of the process, but this and other difficulties, it is hoped, may be overcome in great measure.

A comparison of two methods for the determination of glycerol.

Kind of wine.	Glycerol (in 100 cc.).		Water-insoluble residue in extract from 100 cc of wine.	Tannin in extract from 100 cc of wine.	Percentage composition of extract weighed as glycerol in provisional method.			
	By provisional method.	By oxidation method.			Glycerol by oxidation.	Insoluble residue.	Tannin.	Undetermined residue.
	Grams.	Grams.	Grams.	Grams.	Per cent.	Per cent.	Per cent.	Per cent.
Zinfandel.....	0.6974	0.6128	0.0066	0.0115	87.86	0.95	1.65	9.54
Burgundy.....	.2592	.2226	.0016	.0083	85.88	.62	3.20	10.30
Sherry.....	.9599	.8.94	.0059		88.48	.61		10.91
Port.....	1.1595	1.0064	.0046	.0440	86.79	.40	3.79	9.02
Muscat.....	1.0424	.8900	.0019	.0325	85.38	.18	3.12	11.32
Hock.....	.6947	.6014	.0037	.0010	86.57	.53	.14	12.76
White wine.....	.6440	.5458	.0010		84.75	.16		15.09
White wine.....	.6408	.5498	.0000		85.79	.00		14.21
Port.....	.6682	.5890	.0012	.0135	88.01	.18	2.02	9.79
Sherry.....	.7765	.6960	.0015	.0210	89.63	.19	2.70	7.48
Claret.....	.7310	.6214	.0033	.0053	85.00	.45	.72	13.83
Angelica.....	.6870	.5856	.0014	.0273	85.24	.20	3.97	10.59

THE DETERMINATION OF REDUCING SUGARS IN WINE.

The following criticism of the provisional volumetric method of the association was prepared by R. M. West, St. Paul, Minn.:

The chief objection to the present provisional methods for the determination of reducing sugars in wine (Bul. 107, p. 87) is the length of time necessary for the operation. Many attempts have been made to remedy this defect by the introduction of volumetric methods, which as a rule have been unsatisfactory either through the necessity of preliminary titrations or through the excessive errors caused by the variable conditions under which the copper is precipitated. It was decided after careful consideration that the actual precipitation of the copper by the provisional method could not be modified to advantage, but that the ordinary methods of preparing the sample, in addition to being long and tedious in operation, are the sources of several serious errors. A strong solution of alcohol fails to reduce Fehling solution, and, such being the case, it is apparent that the addition of 25 cc of a 15 per cent solution of alcohol to 120 cc of boiling diluted Fehling solution, from which it would be almost immediately boiled off, could have little or no influence on the precipitation of the copper. Since the provisional method calls for the dealcoholization of the wine previous to clarification, its omission would save at least a half hour without decreasing the accuracy of the result. Furthermore, it has long been realized that the use of lead subacetate as a clarifying agent has been attended with several errors. In the first place, the precipitated solids carry with them small amounts of sugar, and, in the second place, in taking an aliquot portion of the filtrate the volume of the precipitate is not considered. This second error is repeated when the excess of lead is removed with sodium sulphate, and all these errors together, when multiplied by the number of times that the sample has been diluted during the preparation of the solution in which the sugar is determined, may amount to a considerable proportion of the total reducing sugar content of the wine.

It is proposed, then, that the preliminary treatment of the wine consist of only the dilution necessary to obtain a solution containing not more than 1 per cent of reducing sugar, that the copper be precipitated in the usual way, filtered as quickly as possible, redissolved, and determined volumetrically or by electrolytic deposition. Analyses were made on fourteen samples of wine by both the provisional and the proposed new methods, with the results shown in the accompanying table. To make these results more easily comparable, the sample was diluted with the same amount of water for both determinations.

Comparison of methods for the determination of reducing sugars in wines.

Kind of wine.	Total solids in 100 cc.	Reducing sugar in 25 cc of diluted solution (containing not more than 1 per cent of reducing sugar).		Times diluted.	Reducing sugar in 100 cc of wine.	
		By provisional method.	By proposed method.		By provisional method.	By proposed method.
	<i>Grams.</i>	<i>Mg.</i>	<i>Mg.</i>		<i>Grams.</i>	<i>Grams.</i>
Red wine.....	2.40	32.6	31.6	4	0.1304	0.1264
Red wine.....	2.17	27.0	28.1	4	.1080	.1124
Red wine.....	2.18	66.9	62.1	4	.2736	.2484
Sherry.....	4.37	145.9	146.5	16	2.3344	2.3440
Sherry.....	4.77	164.6	162.5	16	2.6336	2.6000
Port.....	12.00	201.9	201.5	40	8.0760	8.0000
Muscat.....	16.22	160.5	169.7	80	13.5000	13.5700
White wine.....	1.72	25.8	26.4	4	.1032	.1056
White wine.....	1.79	21.0	19.4	4	.0840	.0776
White wine.....	1.61	22.5	18.9	4	.0900	.0756
Port.....	13.08	132.2	130.3	80	10.5700	10.4200
Sherry.....	5.47	87.2	85.7	40	3.4280	3.4280
Claret.....	2.34	51.8	59.1	4	.2072	.2364
Anglica.....	12.83	120.5	127.9	80	10.1200	10.2320

EXAMINATION OF THE NATURAL COLORING MATTER IN WINE.

The following statement was contributed by Genevieve Imus, St. Paul, Minn. A complete tabulation of results of tests made on wine colors is given in the Twelfth Biennial Report of the Minnesota State Dairy and Food Department, pages 250 to 253.

During the past two years a considerable amount of time has been given to the examination of the natural coloring matter of wine. The work has not included the detection of coal-tar dyes or other added colors but was intended primarily for the purpose of obtaining on samples of known purity data that would be of value as criteria in future routine analyses. The plan of making all color comparisons and descriptions with reference to reliable standards has been adopted, and to this purpose the color standards employed by Mulliken in his book entitled "A Method for the Identification of Pure Organic Compounds," have been found to be admirably suited. These standards consist of 18 pure colors, and of derived tones, 36 tints, 36 shades, and 12 medium broken colors. They are mounted on cardboard in compact form to facilitate their use in the laboratory. A description of the standards and a discussion of the application of color reactions to the examination of unknown substances are given by Mulliken on pages 230 to 234. In matching colors the best results are obtained if the operator stands with his back to a window and not in direct sunlight. The material to be examined should be held about an inch away from the white cardboard accompanying the color charts and alongside the square opening. The cardboard is moved about until the exposed color matches as nearly as possible the color of the material. A clear day is necessary for satisfactory results.

The tests which are described below were made upon the undiluted wine unless otherwise stated. In tests with the various reagents the resulting colors of the solution have been noted as well as the color of any precipitate which may have been formed. In the solubility tests with amyl alcohol the colors of both the alcohol and resulting wine have been matched on the color chart and recorded.

(a) About 5 cc of the sample are poured into each of six test tubes and the following solubility tests are applied: To each of two portions, one acidified with a few drops of hydrochloric acid and one made alkaline with ammonia, 5 cc of ether are added. To each of two portions, made, respectively, acid and alkaline in the same manner,

5 cc of amyl alcohol are added. The tubes are thoroughly shaken and the liquids allowed to separate. If in any case an emulsion is formed, a few drops of ethyl alcohol are added. Similar tests are made on the remaining portions of the sample without the addition of acid or alkali.

(b) Ten cubic centimeters of the sample are made alkaline with baryta water, shaken with an equal volume of amyl alcohol and, after observing the colors of the two layers as directed, acetic acid is added to a filtered portion of the amyl alcohol layer.

(c) To 50 cc of the wine in a beaker an equal amount of water and a few cubic centimeters of dilute hydrochloric acid are added. In this is placed a piece of white, fat-free wool cloth, about 10 cm square, and the solution is boiled from five to ten minutes. The cloth is removed, washed in a stream of water, and after noting the color the wool is treated with a 2 per cent ammonia solution.

(d) Five cubic centimeters of concentrated nitric acid are added to an equal amount of the wine in a test tube.

(e) To 10 cc of the sample in a test tube 5 cc of a neutral or slightly alkaline mixture of a 10 per cent potassium alum solution and a 10 per cent sodium carbonate solution are added, the mixture is shaken and the precipitate formed is separated by filtering. In like manner the wine is tested with 5 cc of a mixture of 10 per cent aluminum acetate solution made alkaline with 10 per cent sodium carbonate solution. A portion of the sample is also treated with a 10 per cent solution of mercuric chlorid.

(f) To 15 cc of the sample in a test tube are added 3 cc of a 10 per cent solution of lead subacetate. The solution is shaken and filtered. It has been found difficult at times to match the color of the precipitate closely with the color chart and in such cases the color has been designated in general terms such as blue-gray or brown-gray.

(g) About 0.05 gram of pulverized yellow oxid of mercury is added to 20 cc of the sample and the mixture heated to boiling and poured through a double filter. After first noting the color of the filtrate a few drops of hydrochloric acid are added.

(h) To each of two 10 cc portions of the sample in test tubes a few drops of 10 per cent solutions of ferric chlorid and of ferrous sulphate are added, respectively.

Application of the solubility tests described above has shown the natural color in wines to be insoluble in ether under all conditions and in amyl alcohol when the wine is previously made alkaline with ammonia. A second dye was obtained with one wine, but its dull appearance and its reaction with ammonia afforded a ready means of distinguishing the dye from those of coal-tar origin and from vegetable dyes of the lichen group. Hydrochloric, sulphuric, and acetic acids brighten or intensify the color of the original wine, ammonium and sodium hydroxids darken the solution, and ammonia alum produces no change. Tests with chalk steeped in albumen^a gave unsatisfactory results.

In general the following conclusions may be drawn from the results obtained by these tests:

1. Nitric acid darkens the original solution of red wines, but produces practically no change in white wines.
2. The lead subacetate precipitates vary from a pale yellow in white wines to a deep blue-gray in red wines, but a violet or red color is never found in the precipitate from a genuine wine.
3. Some highly colored red wines give a slight coloration upon the addition of hydrochloric acid to the filtrate from the yellow oxid of mercury.
4. Ferric salts give a precipitate, ferrous salts do not.

The following results were obtained by H. V. Frost on a sample of red wine labeled "Vino Vecchio del Chianti, Toscano, Italia." No evidence as to the authenticity of the wine was secured, although it was known that the sample was imported from Italy. The designations as to color correspond to those in Mulliken's chart.

^a U. S. Dept. Agr., Bureau of Chemistry, Cir. 25, p. 17.

Color reactions using different solvents under varying conditions (Frost).

GROUP A.

5 cc wine shaken with—	5 cc solvent.		Hydrochloric acid + 5 cc solvent.		Ammonium hydroxid + 5 cc solvent.	
	Upper layer.	Lower layer.	Upper layer.	Lower layer.	Upper layer.	Lower layer.
Ether.....	Colorless	R-NT.....	Colorless	R-T1.....	Colorless	Black.
Amyl alcohol.....	NR-T2.....	R-NT.....	R-T1.....	R-NT.....	Colorless	Black.
Chloroform.....	Colorless	Colorless	R-NT.....	Colorless	Black	Colorless.
Petroleum ether.....	Colorless	R-S1.....	Colorless	R-NT.....	Colorless	Blackish.
Carbon bisulphid.....	R-S1.....	Colorless	R-NT.....	Colorless	Blackish.	Colorless.

GROUP B.

Determination.	15 cc wine shaken with 3 cc of 10 per cent solution of lead acetate.	10 cc wine shaken with 5 cc slightly alkaline mixture of 10 per cent potassium alum solution and 10 per cent sodium carbonate.	20 cc wine boiled with 0.5 gram pulverized yellow oxid of mercury.
Color of wet precipitate.....	B-BTM.....	BG-S2.....	R-T1.
Color of filtrate.....	VR-T2.....	BG-BTM.....	

GROUP C.

A piece of fat-free white wool cloth, 10 cm square, was boiled five to ten minutes in 50 cc of wine to which were added 50 cc water and a few cubic centimeters of dilute hydrochloric acid. The cloth was removed and washed in a stream of water. The dyed cloth matched OR-T2; treated with strong ammonium hydroxid, it became yellow-green, matching Y-BTM.

THE DETERMINATION OF TOTAL, FIXED, AND VOLATILE ACIDS IN WINES.

The first methods of determining the volatile acids in wines consisted in distilling a measured quantity of the sample to about one-third of its original volume and titrating the distillate; the results must obviously have been too low. The next step appears to have been in favor of an indirect course of procedure whereby the total acids were first titrated, then another portion of the wine evaporated off and the fixed acids titrated in the residue. From the difference between these titrations was calculated the amount of volatile acids. Various modifications of this method came into use,^a in all of which the aim appears to have been to liberate the total volatile acids by a prolonged heating of the extract. That an appreciable change might occur in the extract constituents as a result of such treatment was not at first conceived. At a later date, however, there developed grounds for the belief that certain of the fixed acids disappeared, in consequence of which on titrating the residue there was obtained too small a result; hence the result for volatile acids would be too high. These considerations finally resulted in the abandonment of the so-called indirect methods, and it was again proposed to separate the volatile from the fixed acids by means of distillation and to titrate the volatile acids in the distillate.^a But because the volatile acids pass over only slowly and with difficulty, a simple distillation, as in the determination of alcohol, could obviously not be employed. Following a number of attempts which had been made to devise a successful method, Lindemann,^b in

^a Methods proposed by Kissel, Weigert, Nessler and Barth, and Wolff: Zts. anal. Chem., 1869, 8: 416; 1879, 18: 208; 1883, 22: 166; Repert. anal. Chem., 1883, 1: 213.

^b Zts. anal. Chem., 1883, 22: 516.

1883, described a method of driving out the volatile acids with steam, and this forms the basis of the official methods which for some years have been prescribed in Europe and in America. In essential details the present official methods^a appear to comply with the following procedure:

Fifty cubic centimeters of wine are distilled in a current of steam, in the meantime heating the flask containing the sample until the liquid boils, and regulating the flame so that the volume remains constant. Two hundred cubic centimeters of distillate are collected and titrated with tenth-normal sodium hydroxid, using phenolphthalein as indicator.

Chemists who have had considerable experience with this method must have noticed that it often happens that distillates collected beyond 200 cc show a more or less strong acid reaction. This is the case especially with certain red wines, notably burgundys, ports, and clarets, and wines of the sauterne type. In fact, as the accompanying table shows, it seldom if ever occurs that the first 200 cc distillate contains even a fair approximation of the total volatile acids. In some instances it is seen that by carrying the distillation beyond 200 cc the error is very considerable. For example, following closely the provisional method and carrying the distillation to 400 cc, there are shown the following rates of increase in total volatile acids: In a burgundy, approximately 14 per cent; in two samples of port, respectively, 13 and 14 per cent; in a claret, 13 per cent; and in a white wine, 9 per cent. There occur, indeed, wines in which the volatile acids appear never to become completely exhausted, in which, in fact, the distillate fails to appear permanently neutral even after very prolonged distilling. This phenomenon may be attributed not so much to acids of difficult volatility as to a possible decomposition of the extract constituents under the influence of prolonged heating by the direct action of the flame which the official directions require should be maintained below the flask containing the sample. It would seem, therefore, that 300 cc, or at most 400 cc, of distillate should contain practically all of the volatile acids, and that it may not be necessary or practical to prolong the distillation until the last portions of the distillate are neutral.

Volatile acids in wines (by the present provisional method).

No.	Kind of wine.	Distillates (cubic centimeters tenth-normal sodium hydroxid required to neutralize).									Acid in first 300 cc.
		First 100 cc.	Second 100 cc.	Thirđ 100 cc.	Fourth 100 cc.	Fifth 100 cc.	First 200 cc.	First 300 cc.	First 400 cc.	Total 500 cc.	
1	Claret.....	6.30	2.10	0.60	0.30	0.15	8.40	9.00	9.30	9.45	Per ct. 95.2
2	Zinfandel.....	5.80	1.70	.50	.25	.15	7.50	8.00	8.25	8.40	95.2
3	Burgundy.....	9.25	3.65	1.25	.55	.20	12.90	14.15	14.70	14.90	95.0
4	Sherry.....	4.65	1.55	.50	.25	.10	6.20	6.70	6.95	7.05	93.6
5	Sherry.....	5.60	2.20	1.10	.70	.30	7.80	8.90	9.60	9.90	90.0
6	Port.....	10.70	3.45	1.25	.65	.25	14.15	15.40	16.05	16.30	94.7
7	Port.....	5.45	2.00	.70	.35	.15	7.45	8.15	8.50	8.65	94.2
8	Muscat.....	2.75	1.00	.40	.25	.10	3.75	4.15	4.40	4.50	92.2
9	Hock.....	2.85	.90	.50	.25	.10	3.75	4.25	4.50	4.60	92.4
10	White wine.....	4.45	1.45	.80	.45	.15	5.90	6.70	7.15	7.30	91.7
a11	White wine.....	6.90	2.25	.50	.35	.20	9.15	9.65	10.00	10.20	94.6
b12	Port.....	2.15	.80	.35	.20	.00	2.95	3.30	3.50	3.50	94.3
13	Sherry.....	5.90	2.40	.85	.45	.15	8.30	9.15	9.60	9.75	93.8
14	Claret.....	7.65	2.65	.95	.40	.15	10.30	11.25	11.65	11.80	95.7
15	Angelica.....	4.70	1.65	.60	.30	.10	6.35	6.95	7.25	7.35	94.5
16	Port.....	4.10	1.95	.75	.35	.15	6.05	6.80	7.15	7.30	93.1

^a First 11 samples furnished by California Wine Association, 1906.

^b Last 5 samples from miscellaneous sources.

The results obtained on these 16 samples of wine show that in only four cases did the fifth 100 cc distillate require as much as 0.2 cc of tenth-normal alkali to neutralize; hence, for practical purposes, it may be assumed that the vanishing point of the

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 107, p. 86.

volatile acids occurs when 500 cc of distillate have passed over. On this basis the results show that the proportion of volatile acids collected in the first 300 cc of distillate ranges from 90 to 95.7 per cent, only four samples showing a proportion slightly greater than 95 per cent. Thus, even in the present provisional method of the association, if the entire apparatus be of fixed dimensions and relations in addition to the other conditions stipulated, a large part of the fundamental error is still retained even by carrying the distillation to 300 cc. If the distillation be conducted slowly and the volume of the wine permitted to become too large, an insufficient amount of volatile acids will pass over; if the distillation be too rapid and the volume of wine be permitted to diminish too much, there may occur an overheating and the amount of volatile acids will be too great. Finally, in order to obtain the greatest possible concordance in results, the distillation must be watched from beginning to end with the greatest care.

It has also been recognized that the agreement in results obtained by several distillations on a given sample fails to reach as high a degree as ought to be expected, even in approximately exact determinations. When the distillations are carried to 400 cc, or even until the vanishing point of acidity is fairly reached, the results often fail to agree within reasonable limits. A difference in the results of the titrations amounting to from 0.3 to 0.5 cc of tenth-normal alkali has often been noted; and the differences are commonly far greater at the close of the 200 cc period, amounting to from 0.6 to 0.8 cc in several instances.

Briefly, then, the objections to the present provisional method are:

- (1) The method is complicated, requiring rather elaborate apparatus and tiresome supervision.
- (2) The prolonged heating of the wine by the direct action of the flame doubtless affects in some manner the constitution of the acid ingredients.
- (3) The results do not to a sufficient extent represent the total volatile acids.
- (4) The results are not reasonably concordant in the hands of different persons or even in the hands of a single individual.

Owing to these considerations, various chemists have proposed the abandonment of the direct method of determining volatile acids in favor of an indirect course of procedure. After a prolonged examination of the relative merits of these two general methods, Windisch ^a proposes the following:

Twenty-five cubic centimeters of wine are titrated in the usual manner for total acids, using litmus or litmus paper as indicator. Another 25 cc portion is then evaporated on a water-bath in a porcelain dish to 3 to 5 cc, the residue dissolved in about 25 cc of hot water, the liquor again evaporated to 3 to 5 cc, the residue again dissolved in about 25 cc of hot water, and the liquor evaporated a third time to 3 to 5 cc. Finally, the residue is dissolved in hot water and the fixed acids titrated, using litmus as an indicator. From the difference between these titrations the volatile acids are calculated.

The advantages of this over the present provisional direct method are obvious, and have been adequately demonstrated by Windisch and others. The wine is never heated above the temperature of the water-bath and the volatile acids are undoubtedly all driven out, leaving the fixed acids probably unchanged. Furthermore, the results appear to be reasonably concordant and satisfactory. Various modifications of the indirect method of obtaining the volatile acids have appeared.^b

Sellier ^c has described a simple apparatus which consists of a small wide-neck flask into which is fitted a cylindrical-shaped flask. In the bottom of the latter flask is a

^a Zts. Nahr. Genussm., 1905, 9: 70.

^b Methods proposed by Roos and Mestrezat, Guerin, Curtel, and Robin: *Bul. assoc. chim. suc.*, 1907, 25: 41-49; *J. pharm. chim.*, 1907, 25: 491-492; *Ann. chim. anal.*, 1901, 6: 361; *J. pharm. chim.*, 1904, 19: 531-533.

^c *Ann. chim. anal.*, 1901, 6: 414.

small opening leading inward through a siphon-shaped tube bent back upon itself and terminating at a point close to the bottom. In making the determination, 50 to 60 cc of distilled water are placed in the larger flask, the smaller flask fitted into the wide neck by means of a section of rubber tubing, 10 cc of wine run in and the water heated to boiling. The steam passing through the siphon-tube and through the wine carries out the volatile acids. No appreciable change in the volume of the wine takes place. When the water is reduced to about 5 cc, the flame is removed. On cooling the apparatus the remaining wine liquor is drawn down into the larger flask. The small flask is rinsed out with a little hot water and the two flasks disconnected. The liquor is cooled and the fixed acids are titrated.

This method has been employed in the laboratory of the Minnesota Dairy and Food Department in the analysis of a number of samples of wine and in the investigation of a dozen or more of the common varieties of fruit juices, and has proven satisfactory not only from the standpoint of convenience in manipulation but on account of the fact that results appear to be reliable and concordant. It has been noted, however, that in this method as in others the volatile acids are not collected but are allowed to dissipate into the air, and it has seemed desirable to condense the vapors and titrate the volatile acids in the distillate. By joining a condenser to the flask containing the sample there is provided an apparatus (see fig. 1) whereby may be determined in one operation both the volatile and fixed acids on the same portion of wine.

The statement of the method proposed for total, volatile, and fixed acids is given on page 13.

In the laboratory of the California Wine Association the following method of titration is employed:^a

Ten cubic centimeters of wine are measured into a 500 cc beaker without the addition of water. The wine is well shaken to remove carbon dioxide and titrated directly with fifth-normal sodium hydroxide. In the case of heavy-colored wines, no indicator is used; the coloring matter of the wine indicates the end point of the titration. In the case of white wines, the same method of procedure is followed excepting that a little neutral litmus is added. In titrating light-colored red wines, it may be advisable to add litmus, but the indicator is never used unless absolutely required.

A comparison of the results obtained by the various methods of determining total, fixed, and volatile acids is shown in the accompanying table. Total acids were determined by the California Wine Association method, by the method of Windisch and by the proposed new method based on that given by Breteau. Removal of carbonic acid was assured before any of the methods were attempted. Fixed acids were determined by the method of Windisch, by the method of Sellier, and by the proposed new

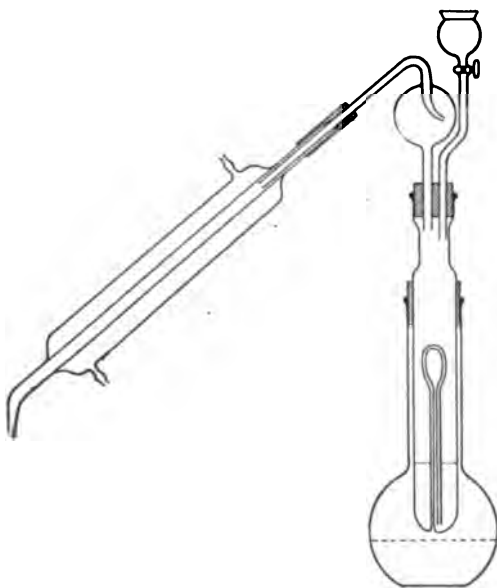


FIG. 1.—Apparatus for determining volatile and fixed acids in wine.

^a According to letter received from C. S. Ash, chemist, California Wine Association.

method of titration after driving off the volatile acids by steam distillation. Volatile acids were determined by the indirect method of Windisch and by the proposed new direct method of distilling by steam. In the Windisch methods the titrations were made using litmus paper as an indicator and in the method of Sellier, as in the proposed new methods, phenolphthalein was used.

Comparison of methods for the determination of total, fixed, and volatile acids in wines.

[Results expressed as cc tenth-normal acid in 100 cc of sample.]

No.	Kind of wine.	Total acids.				Fixed acids.				
		Method of California Wine Association.	Method of Windisch.	Proposed new method.	Calculated from fixed and volatile acids.	Method of Windisch.	Method of Sellier.	Proposed new method.		Calculated from total and volatile acids.
								Using litmus.	Using phenolphthalein.	
1	Angelica.....	4.30	4.16	5.80	5.80	3.32	4.90	3.40	5.00	5.00
2	Claret.....	7.70	7.52	9.60	9.55	6.80	8.00	7.00	8.00	8.05
3	Port.....	5.20	5.60	7.20	7.15	4.36	5.90	5.00	6.00	6.05
4	Riesling.....	6.00	6.36	7.45	7.50	4.80	6.15	5.20	6.10	6.05
5	Sherry.....	4.00	4.50	5.35	5.35	2.96	3.95	3.00	3.90	3.90
a6	Zinfandel.....	8.70	7.92	9.70	9.75	5.64	6.75	5.50	6.75	6.70
b7	Sauterne.....	6.70	7.58	8.55	8.60	5.48	6.40	5.00	6.50	6.45
b8	Bordeaux.....	7.10	8.16	9.15	9.20	6.00	7.30	6.00	6.95	6.90
9	Sauterne.....	7.70	8.20	8.90	8.95	6.00	7.75	6.50	7.30	7.25
10	Port.....	8.90	8.44	9.55	9.60	3.60	4.80	3.70	4.40	4.40
11	Sherry.....	5.50	5.80	6.60	6.60	7.40	9.60	8.20	9.20	9.20
c12	Claret.....	5.60	10.60	11.90	11.90	4.40	5.50	4.50	5.20	5.20
d13	Port.....	6.80	6.30	7.10	7.10	3.60	5.70	4.00	4.80	4.75
14	Sherry.....	5.80	6.40	7.20	7.25	6.20	7.90	6.40	7.70	7.60
15	Claret.....	8.50	8.40	9.50	9.60	6.14	8.20	6.55	8.10	8.05

No.	Kind of wine.	Volatile acids.				Ratio of volatile to fixed acids.	
		Method of Windisch.	Proposed new method.		Calculated from total and fixed acids.	According to method of Windisch.	According to proposed new method.
			Using litmus.	Using phenolphthalein.			
1	Angelica.....	0.84	0.70	0.85	0.80	1 : 3.95	1 : 6.25
2	Claret.....	1.72	1.40	1.55	1.60	1 : 3.95	1 : 5.16
3	Port.....	1.24	.95	1.15	1.20	1 : 3.51	1 : 5.21
4	Riesling.....	1.56	1.15	1.40	1.35	1 : 3.07	1 : 4.35
5	Sherry.....	1.54	1.20	1.45	1.45	1 : 1.92	1 : 2.69
a6	Zinfandel.....	1.94	1.30	1.85	1.80	1 : 2.90	1 : 3.69
b7	Sauterne.....	2.68	2.10	2.70	2.65	1 : 2.04	1 : 2.40
b8	Bordeaux.....	2.20	1.70	2.00	1.95	1 : 2.73	1 : 3.47
9	Sauterne.....	2.44	2.00	2.30	2.25	1 : 2.46	1 : 3.17
10	Port.....	2.20	1.90	2.20	2.20	1 : 1.64	1 : 2.00
11	Sherry.....	3.20	2.40	2.70	2.70	1 : 2.31	1 : 3.41
c12	Claret.....	1.90	1.60	1.90	1.90	1 : 2.32	1 : 2.74
d13	Port.....	2.80	2.00	2.45	2.40	1 : 1.28	1 : 1.96
14	Sherry.....	2.20	1.60	1.90	1.80	1 : 2.82	1 : 4.05
15	Claret.....	1.78	1.40	1.65	1.60	1 : 3.44	1 : 5.06

a First six wines furnished by the California Wine Association, 1908.

b 7 and 8 obtained from local dealers, 1908, St. Paul, Minn.

c 9 to 12, inclusive, from a Rochester, N. Y., wine company, 1908.

d 13 to 15, inclusive, from a Norfolk, Va., wine company, 1908.

The results obtained by the California Wine Association method were not satisfactory, the end-point of the titrations being, in most instances, very uncertain. In the angelicas, ports, and sherrys especially, much difficulty was experienced

in carrying out the titrations, and the results were scarcely better when litmus tincture was used. In titrating according to the Windisch methods the point of neutrality was judged to be attained when a small drop of the liquor placed on delicate blue litmus paper just ceased to produce a perceptible red. There appeared to be decided disadvantages in using litmus paper, and the use of litmus tincture even in a clear distillate is open to serious objections, which will be stated presently. In colored wines especially the difficulties were very great, and it was found well-nigh impossible at times to devise a means whereby to judge with reasonable certainty the true end-point of the titration. It was found, however, after considerable practice, that fairly concordant results were obtainable by this method in the majority of instances. Phenolphthalein, on the other hand, while not entirely objectionable, was found to give far greater satisfaction. While it was not always convenient to titrate on the undiluted sample, especially in the case of wines containing more or less natural coloring matter, it was found to be entirely permissible, as in the titration of cider vinegars, to dilute with boiled distilled water in order to carry out a successful titration with phenolphthalein. It has been shown that the end-point of a titration can be very accurately judged, even in a deeply-colored wine, and that the addition of water to the extent of 100 or 200 cc does not introduce a serious error in the result. As in a cider vinegar, the change in the color of a wine occurs at a much earlier stage than the change in the indicator and there is never a serious difficulty in safely judging the end point.

As already pointed out, the results shown in the first column of figures are at best only rough approximations. In the majority of instances it was observed that when litmus paper was used the titrations were carried somewhat beyond the point of neutrality which seemed to be indicated by the change in the natural coloring matter of the wine. It is also noted that the results obtained by the titrations employing litmus were uniformly much lower than the results obtained with phenolphthalein. This is true not only in the titrations of total and fixed acids, but also in the direct titrations of the volatile acids. On the basis of the results obtained with phenolphthalein, litmus indicates approximately from 77 to 92 per cent of the total acids and from 58 to 85 per cent of the fixed acids. Doubtless there are theoretical reasons underlying these differences, and the question may well be raised as to whether chemists have given due attention to these considerations in choosing indicators for titrating the acids in wines.

In the first place, there appears to be little justification for the practice adopted by some chemists of employing the natural coloring matter as a correct indicator in titrating either the total or fixed acids. Little of value is known regarding the action of the oenocyanin or other coloring substances in the presence of acids or alkalies, and it is certain that such substances have not been recommended in the titration of any of the common acids. In the case of litmus also there are some important considerations which should bar it as an indicator for wines as well as fruit products in general. Litmus is not recommended for titrating such acids as tartaric, acetic, tannic, succinic, or malic. In titrating tartaric acid with this indicator, the change is gradual and the end-point indistinct, while in titrating acetic acid, the acetate of sodium formed is alkaline to litmus and tends strongly to hasten the end-point. On titrating solutions of tannic acid, a change takes place almost immediately on beginning the titration, and only a small proportion of the actual acid is indicated. Phenolphthalein, on the other hand, is a very satisfactory indicator with all these acids, and, with the exception of tannic acid, the theoretical amount of acid is obtained. About 80 per cent of tannic acid is indicated, but the total acid is obtained after boiling with a measured small amount of tenth-normal hydrochloric acid.

As a means of shedding some light on the differences occurring in titrating wines with the two indicators, the determinations shown in the following table have been carried out:

Comparison of litmus and phenolphthalein as indicators in titrating some of the organic acids existing in wines.

Acid.	Description.	Grams in 100 cc.	Normal acid in 100 cc.			Per cent acid indicated.	
			Calculated.	Using phenolphthalein.	Using litmus.	Using phenolphthalein.	Using litmus.
			cc	cc	cc		
Tartaric.....	Elmer and Amend.....	0.4000	5.33	5.35	5.10	100.3	95.6
Acetic.....	Mallinckrodt's 99 per cent.....	.4040	6.67	6.70	6.35	100.4	95.2
Potassium bitartrate.....	Elmer and Amend.....	.4000	2.12	2.10	1.90	99.0	89.6
Tannic.....	J. T. Baker Chemical Co.....	.5000	2.79	2.20	.40	78.8	14.3
Tannic.....	After boiling with dilute hydrochloric acid.			2.80	.40	100.3	14.3
Average (omitting last item).						94.4	73.6

The results shown for volatile acids by the Windisch method (p. 22) are somewhat higher than those obtained by the proposed new method, using phenolphthalein. Such discrepancies, however, lose their significance when it is considered that in the determination of volatile acids by the indirect method not only are the results of the titrations employing litmus as indicator incorrect, but the titrations of total and fixed acids are not made under comparable conditions. While it is unquestionably true that the volatile acids may be completely driven off by repeated evaporation in an open dish, it does not follow that the results obtained by means of the two titrations are correct. It is conceivable that important changes may occur during the prolonged heating of the wine in order to reduce the material a third time to a pasty consistency. At any rate, we have no positive knowledge that the so-called fixed acids occurring in the final residue represent the actual fixed acids in the original wine. A titration of the residue may suffice as an indication of the acids remaining after driving off the volatile constituents by prolonged heating, but to employ the result of such a titration as a factor in the calculation of the actual volatile acids appears to be an unwarranted proceeding.

In expressing results of analysis the orthodox custom appears to be to calculate the fixed and total acids as tartaric and the volatile acids as acetic. It is impossible to concede any advantages in favor of this custom. It may be safe to assume that in wines the fixed acids are in the main tartaric and the volatile acids acetic; but, even on such assumptions, the results are strictly erroneous and not readily comprehended. Such a method applied to the various fruit juices and ciders would fail to give significant results in practically all cases, and the case is still worse when one adopts the method of calculating the acids as sulphuric. Instead of these conventional methods it has been found better to adopt the plan of expressing all results for total, volatile, and fixed acids in terms of the number of cubic centimeters of normal acid in a definite measure, say 100 cc, of wine. There will then be afforded results which can be readily compared and comprehended. Furthermore, in case it be required to calculate results in terms of any particular acid, such an operation can easily be carried out.

RECOMMENDATIONS.

(1) The standard temperature for the determination of specific gravity should be changed to 20°C. A statement of reasons for this change seems to be unnecessary, as the matter has been fully discussed by others, and many chemists have for some time

adopted the custom of making determinations at a temperature not far from that of the average laboratory. If the alcohol tables can be revised in accordance with a standard temperature of 20° C. for specific gravity determinations, a very useful service will be performed, especially in the interest of industrial and food chemists.

(2) The method for glycerol should be made a subject for special study. Experience has shown that it is possible not only to increase the accuracy of the method but to shorten the time of the operation. As the provisional method now stands, it appears to be rather tedious, and there are too many opportunities for error. A large error undoubtedly occurs during the evaporations as well as during the repeated extractions. Also, it appears that the residue weighed as glycerol is far from being pure.

(3) The present methods for determining total, fixed, and volatile acids are exceedingly faulty. The method for volatile acids, especially, fails to give results anywhere near the truth. The difficulty lies not only in the collection of 200 cc distillate but in the operation, which is cumbersome and unreliable. The use of litmus in the titrations of total and fixed acids is open to criticism, as that indicator fails to show all of the acids. A study of the proposed new methods is recommended.

(4) A more comprehensive scheme for the examination of the natural coloring matter of wines is required. Attention is called to the use of standard color charts as a means of obtaining comparable results in the hands of different persons. It is recommended that the association make a special study of the character and properties of the coloring matters existing in genuine wines.

REPORT ON BEER.

By H. E. BARNARD, *Associate Referee*.

Mr. Barnard, referee on beer, reported that no cooperative work on the subject had been done, and made the following statement in regard to the condition of the methods:

Two years ago I presented beer methods which have since been adopted as provisional. I have been working with those methods since that time and find no special necessity for changing them. For that reason I have not made a special report on beer. Much work, however, seems to be necessary if we must determine the different kinds of beer, and I would only suggest to you the necessity for a careful study of methods of beer analysis with special reference to the adoption of some method which will enable us to tell more accurately than is at present possible whether or not beer is brewed from all malt, or part malt, or from malt substitutes.

REPORT ON DISTILLED LIQUORS: COOPERATIVE TEST OF METHODS FOR THE DETERMINATION OF FUSEL OIL.

By L. M. TOLMAN, *Associate Referee*.

The cooperative work undertaken this year was a comparison of the present Allen-Marquardt method, as given in Bulletin 107, revised, page 98, and a proposed modification worked out by the associate referee and his assistants. The modification was based on the determination of the amount of bichromate reduced in the oxidation of the higher alcohols. This method eliminates the distillation of the acids, which the experiments made have shown are not completely distilled off. In order to test this modified method (for details see paper, p. 206) a series of samples was prepared containing varying amounts of pure amyl alcohol (boiling point 131° C.) in approximately 50 per cent by volume ethyl alcohol, and the samples sent to eighteen different laboratories, asking for a comparison of the modified method with the present method as given in Bulletin 107. Eleven reports were received, and the following table gives

the results, the percentage yields being calculated from the grams of amyl alcohol per 100,000 of 100-proof alcohol, as determined by each method.

Comparison of the Allen-Marquardt method and the proposed modification for the determination of fusel oil, using varying amounts of amyl alcohol.

Collaborator.	0.050 gram.		0.100 gram.		0.150 gram.		0.200 gram.		0.250 gram.		0.350 gram.	
	Allen-Marquardt method.	Modification.	Allen-Marquardt method.	Modification.	Allen-Marquardt method.	Modification.	Allen-Marquardt method.	Modification.	Allen-Marquardt method.	Modification.	Allen-Marquardt method.	Modification.
	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.
Detroit laboratory	99.8	102.0					78.03	111.9	77.74	97.69	100.0	85.14
New York laboratory	100.0	122.0	78.7	105.0	110.0	130.4	74.1	78.34	62.7	85.69	62.3	88.3
Philadelphia laboratory	82.0	113.0	72.11	86.0	73.8	85.4	78.5	88.06	74.5	87.7	79.7	87.24
Portland laboratory	56.0	106.4	99.70	105.1	80.4	96.6	83.4	95.02	79.4	104.0	76.7	87.7
Sunnybrook Distilling Co.	120.0	125.9	108.8	104.5	100.0	114.9	87.06	108.6	87.86	85.64	80.6	81.68
St. Paul laboratory	103.0		82.64				78.0		83.22		69.06	
Washington laboratory	67.3	134.2	72.1	97.1	62.2	91.5	65.2	86.2	68.3	88.5	68.8	84.6
Galveston laboratory ^a	87.20	87.24	79.28	82.14			80.0	79.90	81.36	78.60	72.06	75.52
San Francisco laboratory ^a					42.8	63.1	50.44	74.2	53.74	80.0	50.9	73.22
Seattle laboratory ^a	75.6	70.4					61.62	66.60	70.16	62.14	61.86	68.19
Boston laboratory ^a	84.40	145.7		119.1	76.04	124.5	76.36	131.4		121.0		110.3
Average	89.7	117.2	85.7	99.5	85.3	103.7	77.7	94.7	76.2	91.5	76.7	85.8

^a Excluded from average.

Some of the results obtained at laboratories which had not had experience in operating the method varied markedly from the other figures and are omitted from the average. The averaged results on the various amounts by both methods are plotted, using as the abscissa of the curves the amount of amyl alcohol used in grams per 100,000 of

proof spirit and as ordinates the average percentage yield.

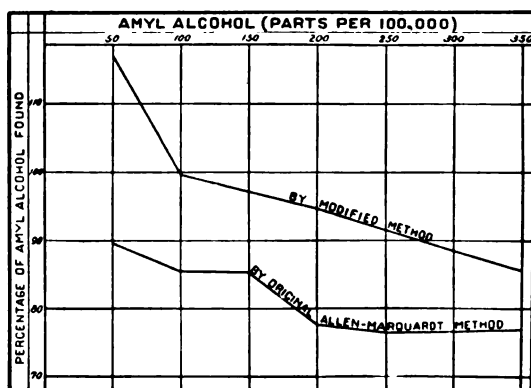


FIG. 2.—Graphic of collaborators' results on amyl alcohol by the Allen-Marquardt method and the proposed modification.

This curve (fig. 2) shows that the new modification gives uniformly higher results, indicating a regular loss in the old method. This loss is undoubtedly largely due, as is shown by the experiments, to the failure to drive over all of the volatile acids in the distilling method unless a much larger amount of water is distilled than that prescribed in the present provisional method. A very much higher yield of acids

is obtained by carrying the distillation much further, as is pointed out in the supplementary paper submitted on this subject (p. 206). There is also shown a uniformly increasing loss by both methods as the per cents of amyl alcohol increase. This is doubtless due to the method of extraction, as a 100 per cent yield can be obtained in the oxidation part of the method, as was demonstrated in the experimental work on the determination of the factor 0.001773 (see p. 210). From these results it is evident that a higher yield is due to the more correct estimation of the higher alcohols present in the

carbon tetrachlorid extract. The curve also shows that the collaborators obtained more uniform results from the new modification than from the method as originally stated. It may be concluded, therefore, that—

- (1) The modified method gives higher and more uniform results.
- (2) It eliminates a tedious and inaccurate distillation.
- (3) It is quicker and gives an opportunity to make check titrations on the same sample.

RECOMMENDATIONS.

As a result of this year's work, including that reported on page 206, it is recommended—

- (1) That this modification of the Allen-Marquardt method be adopted as a provisional method (see p. 210).
- (2) That in the present method a second washing with sodium sulphate be prescribed.
- (3) That the method for determining the water-insoluble color be adopted as a provisional method (see p. 207).
- (4) That the method for the determination of amyl insoluble color be adopted as provisional (quantitative Marsh test method, p. 206).
- (5) That the Roese method given in Bulletin 107, page 97, be dropped as a provisional method on account of the entirely unsatisfactory results obtained with it in the past two or three years.

Mr. Tolman called on Mr. Fischer, of the Bureau of Standards, who spoke in regard to the necessity of unifying the alcohol tables. He called attention to the fact that two tables are now in use by the Treasury Department and a third by the Association of Official Agricultural Chemists. The disadvantages to chemists and practical workers from such a condition of affairs being obvious, it was strongly recommended that the association take some action in the matter. A table based on the calculations of Mendeléeff was recommended by Mr. Fischer. The question of temperature was also discussed, and the whole matter was temporarily referred to Committee C on recommendations of referees, the chairman naming the following members to serve on this committee: Messrs. Tolman, Winton, Hortvet, Bartlett, and Jaffa.

REPORT ON VINEGAR.

By CHARLES H. HICKEY, *Associate Referee.*

The work which has been done by or reported to the referee deals largely with the lead number for pure cider vinegar and other pure vinegars. The cider vinegars used by the referee were made by the old-fashioned, slow process. The method employed is similar to the one by which Winton and Kreider^a analyzed maple products, with some modifications to make it applicable to vinegar. The number of grams of lead precipitated by 100 cc of vinegar is taken as the lead number. Other data are included to make the results more complete. The method as modified by the referee is as follows:

Pipette 25 cc of vinegar into a 100 cc flask; add 5 cc of a standard lead subacetate solution and dilute to 100 cc. Let stand at least one hour, then filter and pipette out 50 cc of the clear filtrate. To this add 10 cc of dilute sulphuric acid and 100 cc of 95 per cent alcohol; let stand over night; filter through a porcelain gooch crucible; wash

^a J. Amer. Chem. Soc., 1906, 28 : 1204.

with 95 per cent alcohol; dry at a moderate heat for a few minutes, cool and weigh. Calculate the amount of lead in the precipitate (factor 0.6829) and subtract this from the amount in 2.5 cc of the standard solution as determined on a blank test, and divide the remainder by 0.125, thus obtaining the lead number.

The standard lead subacetate used in this work is prepared as follows: Dilute a U. S. P. lead subacetate solution until the specific gravity is 1.25; to one part of this add four parts of water and filter. If the solution becomes cloudy, filter before using, and determine its strength frequently. The referee found that the strength changed but little.

Mr. E. M. Bailey of the agricultural experiment station at New Haven, Conn., has reported work which he did independently on different kinds of samples of vinegar of known purity. He includes other data in his results, especially those on testing a recent method for the determination of malic acid which was formerly applied to maple products.^a He found that by using this method more malic acid can be recovered than by the old calcium chlorid method. The method for determining the lead number is as follows:

Measure 50 cc of vinegar into a 100 cc flask, add 25 cc of lead subacetate (dilute solution used by Winton and Kreider) make up to the mark and filter. To 10 cc of the filtrate add 1 cc of concentrated sulphuric acid, 40 cc of water and 100 cc of 95 per cent alcohol. Filter after 12 hours, ignite, and weigh.

The amount of lead in the blank test is determined by diluting 25 cc of the lead subacetate solution to 100 cc; 10 cc are taken out and the lead number determined as in the method just given.

The modified method for malic acid as applied to vinegar is as follows:

To 10 cc of vinegar add an equal volume of water, 3 cc of a 10 per cent solution of calcium acetate, and 180 cc of 95 per cent alcohol. Heat on the steam bath for from 20 to 30 minutes, stirring vigorously at intervals to insure a clear supernatant liquid. Filter on 589 S and S paper, wash with 85 per cent alcohol, and ignite. Dissolve in excess of tenth-normal hydrochloric acid (10 cc) by gentle boiling, and continue to boil for about 10 minutes. Cool and titrate with tenth-normal sodium hydroxid, using methyl orange as indicator.

The results of the work of the referee and those of Mr. Bailey appear in the accompanying table. This shows the variation in the amount of lead precipitated by the different vinegars. In the case of malt vinegar, the results tend to run high; while those of the sirup and distilled vinegar are very low.

It should be noted that in the case of malic acid determinations, which are, of course, not properly such on malt and sirup vinegar, misleading results may be obtained and, in the case of a suspected sample, the malic acid determination would have to be confirmed by the procedure recommended by Leach and Lythgoe.^b

In comparing the figures for malic acid, phosphates, and the lead number as worked out by Mr. Bailey, it is his opinion that a closer relation exists between the phosphate content and the lead number than between the malic acid value and the lead number. The three highest lead numbers are associated with the three highest total phosphate values; the same is true of the three lowest figures in each case. That this does not follow, however, in the case of lead numbers and malic acid values would seem to indicate that the precipitate produced on adding lead acetate to vinegar is due rather to the phosphates than to the malates. This is in accordance with the statement of Leach and Lythgoe^c that the precipitate produced by lead acetate is not entirely due to malic acid. Tolman and Le Clerc^d are also of this opinion.

The other data for pure cider vinegar, included in the table, are fairly typical, and in addition to the old figures the new ones for the lead number are of interest.

^a J. Amer. Chem. Soc., 1908, 30 : 1285.

^b J. Amer. Chem. Soc., 1904, 26 : 379.

^c J. Amer. Chem. Soc., 1904, 26 : 380.

^d U. S. Dept. Agr., Bureau of Chemistry, Bul. 99, p. 89.

Analyses of samples of vinegar of known purity.

C. H. HICKEY.

Number of sample.	Character.	Solids.	Acid.	Ash.	Reducing sugars (dextrose).		Reducing sugars in solids.	Polarization (200 mm Vent-tube).	Malic acid.	Phosphoric acid (per 100 cc).		Lead number for 100 cc.
					Di-rect.	In-vert.				To-tal.	Sol-uble.	
		P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	°V	P. ct.	mg.	mg.	
1	Cider vinegar.											0.166
2	do											.134
3	do	1.44	5.16									.087
4	do	2.69	8.70									.145
5	do	1.50	5.06	0.19								.106
6	do	1.70	4.57	.38	0.13	0.13	7.6	-1.10				.106
7	do	2.18	4.60	.35	.24	.24	11.4	-1.87				.112
8	do	1.75	5.62	.38	.12	.10	6.3	-1.54				.095
9	do	1.98	5.78	.32	.21	.19	10.1	-0.88				.114
10	do	2.04	6.30	.37	.22	.20	10.2	-1.43				.103
11	do	2.10	7.84	.48	.11	.11	5.2	-1.21				.114
12	do	1.86	6.24	.40	.12	.12	6.5	-0.88				.103
13	do	1.76	5.74	.30	.14	.13	7.7	-1.10				.109
14	do	1.50	3.00	.34	.09	.09	6.0	-0.77				.098
15	do	1.94	5.80	.46	.14	.18	8.3	-0.88				.129
16	do	1.73	4.84		.16	.16	9.3	-0.88				.106
17	do	1.35	4.60	.32	.10	.10	7.4	-0.88				.087
18	do	1.60	4.72		.12	.11	7.2	-1.10				.103
19	do	1.80	7.28	.40	.13	.11	6.7	-1.08				.076
20	do											.109

E. M. BAILEY.

21	Cider vinegar.	1.50						0.073				0.075
22	do	2.52			1.01	1.04	40.0	.129	15.1	10.2		.116
23	do	2.34			.95	.99	41.4	.121	15.9	9.0		.088
24	do	2.34			.50	.53	21.3	.411	16.6	6.7		.148
25	do	2.77			.86	.93	32.5	.296	26.9	13.8		.174
26	do				.87	.89		.198	46.1	39.9		.290
27	do				.62	.64		.221	18.4	10.8		.122
28	do	3.45			1.12	1.14	32.3	.399	30.0	16.9		.220
29	Malt vinegar ^b	2.40	5.74	0.06								.434
30	do	2.79			.90	.97	33.3	.230	106.2	17.8		.548
31	do	1.69			.08	.09	5.3	.120	31.5	14.6		.158
32	Distilled white.	0.25			.01	.02	8.0	.016	5.9	3.3		.018
33	Sirup.	1.17			.19	.20	17.0	.069	12.8	1.8		.021
34	do. ^b	1.60	4.48									.015

^a Incompletely acetified^b Not analyzed by E. M. Bailey.

REPORT ON FLAVORING EXTRACTS.

By E. M. CHACE, *Associate Referee*.

No report on flavoring extracts was submitted by the referee last year, owing to the fact that only a very limited amount of work was done, and the report this year includes also the work submitted by collaborators in 1907.

WORK OF 1907.

In 1907 the following samples were sent to collaborators, with the usual instructions.^a

No. 1. Blank containing no lemon oil or citral.

No. 2. Alcohol 1,900 grams, lemon oil 100 grams.

No. 3. Lemon oil used in preparing No. 2.

No. 4. Alcohol 2,000 grams, vanillin 2 grams, coumarin 2 grams, acetanilid 1 gram.

No. 5. Extract prepared from Mexican vanilla beans by the U. S. P. method.

^a The methods for citral were the same as those reported for 1908, see page 32; for vanilla methods see Bul. 107, p. 156.

Reports were received in all from seven collaborators, and the results are tabulated as follows:

Citral determinations in lemon extracts and oil, 1907.

Collaborator.	Sample 1.	Sample 2.	Sample 3.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
A. P. Sy, Washington, D. C.	None	0.16	5.23
B. H. Smith, Boston.	Trace	.300	6.12
C. S. Brinton, Philadelphia.	Not reported	.322	Not reported
A. F. Seeker, New York.	0.01	.226	4.61
Do. a.		.209	4.50
L. A. Brown, North Dakota.	.00	.229	5.41
W. A. Syme, North Carolina.	.027	.126	3.50

a Using ice water bath.

COMMENTS OF THE ANALYSTS.

A. P. Sy: Sample No. 2—oil globules had separated in original sample. Shook well before making determination.

B. H. Smith: These were the first samples personally examined by this method and I have not found time to repeat the work as was intended before reporting results.

C. S. Brinton, and T. F. Pappe: We desire to state several points which will probably have a bearing on the value of these results: First, the metaphenylene diamine hydrochlorid at our disposal showed signs of being somewhat decomposed and the aldehyde-free alcohol, as a result, gave a quite marked coloration with the reagent. Second, although we allowed several days to elapse before making up to volume our fuchsin sulphur dioxide reagent, it did not become colorless but showed a deep lemon yellow tint.

A. F. Seeker: In laboratories where constant temperature baths are not at hand and when only occasional citral determinations are required, it will be found much more convenient to use ice water for immersion of reagents and colorimeter tubes. Provided the standard and the unknown solutions were subjected to the same conditions it was thought that the results might be as accurate. To test this, the constant temperature bath was used in one set of determinations and in the other an ordinary ether can filled with water in which a piece of ice was constantly kept. The latter requires no watching and the color develops less rapidly, making it possible to read a solution containing three milligrams of citral without difficulty. At 15° the color developed by three milligrams is a little too intense. Results at 15° are slightly higher.

In preparation of aldehyde-free alcohol, it was found that three grams of metaphenylene diamine hydrochlorate per liter *** was sufficient provided the alcohol was boiled for eight hours and allowed to stand over night. ***

To ascertain to what extent the aldehyde in commercial spirits used for making up extracts might affect the citral determinations, a sample of ordinary 95 per cent alcohol was run in the same manner as an extract. It showed aldehyde equivalent to 0.031 gram citral. Results obtained with extracts may thus be a little higher than the truth for this reason.

Linwood A. Brown: In the determination of citral by the fuchsin method, the greatest objection to it is in obtaining alcohol perfectly free from aldehydes. The Dunlap method failed to give a perfectly aldehyde-free alcohol even after three times on the same samples of alcohol, i. e., the alcohol was subjected to the method three successive times.

The metaphenylene diamine hydrochlorid method gives the best results for this determination.

W. A. Syme: Commenting on the method for lemon extracts, I would say that the method for purifying the alcohol (with metaphenylene diamine) did not yield an alcohol that would not produce a color with fuchsin solution on two trials. This lessens the accuracy of the work. I would suggest that other methods of preparing alcohol be studied and that other solvents be tried.

A glance at the table is sufficient to show that the results obtained in 1907 were practically of no value. The discordant figures on sample No. 2 are in part explained by the fact that this extract was made up in 85 per cent alcohol and it was found later that globules of oil had separated and were floating on the surface. This fact is noted in the comments of Mr. Sy, who analyzed the sample some time after it had been made up.

The only other explanation offered, is that the analysts were not familiar with the method. So far as is known Mr. Seeker is the only collaborator who had had any such

experience and his results on samples Nos. 2 and 3 are very close to the theoretical figures. The following results were obtained on samples Nos. 4 and 5:

Analyses of vanilla extract, 1907.

Collaborator.	Sample No. 4.			Sample No. 5.
	Vanillin.	Coumarin.	Acetanilid.	Vanillin.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
J. M. Bartlett, Maine	0.122	0.079	0.056	a 0.133
A. L. Nehls, Illinois	b .244	.075	.045	.130
A. P. Sy, Washington, D. C.	.083	.076	.022	
T. F. Pappe, Philadelphia.....	.098	.080	.031	.103
J. C. Olsen, New York	.112	.066	.016	.145
L. A. Brown, North Dakota.....	.106	.082	.021	.112
Theoretical amount present.....	.0997	.0997	.0498	
Average	.104	.076	.032	.125
Greatest difference above amount present.....	.022		.006	
Greatest difference below amount present.....	.014	.034	.004	
Difference between maximum and minimum.....	.039	.014	.040	.022

a Also reported 0.014 per cent coumarin in this sample.

b Omitted from averages.

COMMENTS OF THE ANALYSTS.

A. L. Nehls: It would be much more accurate to determine the specific gravity of the solution, and pipette off a known volume for analysis. It was found in this work that solution No. 4, standing in the beaker on the balance pan for five minutes lost 10 mg. This means that under ordinary conditions the third decimal place in the result is meaningless.

In this laboratory we use a new test for coumarin which was originated by Doctor Bryan. It depends on the fact that a drop of alcoholic potash solution containing 20 grams of potassium hydroxid to a liter will, when placed on crystals of coumarin, give a lemon yellow color. This color is very intense, but disappears rapidly. Neither vanillin nor acetanilid is affected by the reagent. The test is applied directly on the crystals by using a glass rod for a dropper, and the results have always been much more satisfactory than those obtained by any other test, because of its great delicacy. Both vanillin and coumarin give a yellow solution on standing for some time, but the crystals of vanillin remain uncolored until dissolved, while the coumarin crystals are intensely colored.

The color tests for acetanilid are not as satisfactory as they might be. In this work we found the polarizing microscope of the greatest use. There is little danger of confusing either of the three crystals which are likely to occur in a vanilla extract. The discrepancies in the results for vanillin are quite usual where the crystals are weighed directly from an ether solution. They seldom come out well, usually being colored, often nearly black. This can be remedied by another extraction, not with ether.

A. P. Sy: On testing this residue for acetanilid, (Ritser's tests) no reaction for same could be obtained. As only 25 grams extract are taken, the residues actually obtained were 0.0029 gram and 0.0026 gram for the duplicates. Using 4 mg of pure acetanilid, no reaction could be obtained by Ritser's tests as given in Bul. 107. Using chlorin water (U. S. P.) instead of a solution of chlorinated lime (1 : 200) a good reaction was obtained with 4 mg acetanilid; 2 mg gave fair test. The chlorin water is mixed with the acetanilid; a pink color forms in a few seconds, changing gradually to a purple and finally to a blue.

J. C. Olsen: *Vanillin:* The residue of the ether extraction for vanillin almost invariably contains a large amount of resin and other impurities. It has always been our custom to extract the vanillin with petroleum ether and deduct the residue from the weight of impure vanillin. It will be noted that the difference in results is considerable. In 18 determinations the impurity with the vanillin has varied from 2 to 30 mg, the average being 10.4 mg.

We have also found that an easier drying residue of vanillin has been obtained by extraction from the 2 per cent ammonia solution with chloroform.

Coumarin: For the separation of coumarin and acetanilid we have been unable to obtain petroleum ether with a boiling point 30-40° C. We have used gasoline, 86° B. On fractionating this naphtha, fractions boiling at 35-40, 40-45, 45-50, 50-60, etc.,

have been obtained. It has been our experience that the higher boiling fractions extract coumarin as well as vanillin fully as well as the lower boiling fractions.

Acetanilid: According to the official method this substance is to be looked for with the vanillin only when it has been found with the coumarin. In one of these three analyses of No. 4 reported all of the acetanilid was found with the vanillin.

The figures given by Mr. Olsen on vanillin by extraction with petroleum were as follows: No. 4, 0.080; No. 5, 0.054, from which it would appear that the extraction was prolonged sufficiently.

Linwood A. Brown: Sample No. 5: The vanillin in this sample was somewhat impure owing to coloring matter from which I was unable to purify it.

The result would seem to show that as far as vanillin is concerned the method is satisfactory. The average on both vanillin and coumarin, however, indicates that some of the latter is weighed as vanillin. The coumarin figures are uniformly low, as are those for acetanilid, with one exception. One collaborator reports entire failure of the Ritsert's test for acetanilid as given in the provisional methods, and suggests a modification.

WORK OF 1908.

The work for 1908 was confined to the colorimetric method for the determination of citral in lemon extracts. Fifteen sets of samples were sent out to collaborators who had previously worked with the method, and reports have been received from twelve. As the method had been rather severely criticised by some of the members of the American Extract Manufacturers' Association, they were invited to name two collaborators, and selected Mr. Edward Kremers, of the Wisconsin State College, and Mr. Baer, of St. Louis. Samples were sent to both, and Mr. Kremers forwarded his set to I. W. Brandel, of the University of Washington. The following description of the method to be used was sent to each collaborator:

DETERMINATION OF CITRAL IN LEMON EXTRACT.

Reagents.

Aldehyde-free alcohol.—Allow alcohol (95 per cent by volume) containing 5 grams of metaphenylene diamine hydrochlorid per liter to stand for twenty-four hours with frequent shaking. (Note, nothing is gained by previous treatment with potassium hydroxid.) Heat under a reflux cooler for at least eight hours, longer if possible (often twenty-four hours are necessary), allow to stand over night and distil, rejecting the first 10 and last 5 per cent which come over. Store in a dark, cool place in well-filled bottles.

Fuchsin solution.—Dissolve one-half gram of fuchsin in 250 cc of water, add an aqueous solution of SO_2 containing 16 grams of the gas and allow to stand until colorless, make up to one liter with distilled water. This solution should stand twelve hours before using and should be discarded after three days.

Standard citral solution.—One milligram of c. p. citral per cubic centimeter in 50 per cent by volume aldehyde-free alcohol.

Apparatus.

A cooling bath.—To be kept at from 14°C. to 16°C. The aldehyde-free alcohol, fuchsin solution, and comparison tubes are to be kept in this bath.

Colorimeter.—Any form of colorimeter using a large volume of solution and adapted to rapid manipulation may be used.

The comparison may also be made in Nessler or Hehner tubes.

Manipulation.

Preliminary determination.—Weigh in a stoppered weighing flask approximately 25 grams of extract, transfer to a 50 cc flask and make up to the mark at room temperature with aldehyde-free alcohol. Measure at room temperature and transfer to a comparison tube 2 cc of this solution, add 25 cc of the aldehyde-free alcohol (previously cooled in the bath) then 20 cc of the fuchsin solution (also cooled) and finally make up to the 50 cc mark with more aldehyde-free alcohol. Mix thoroughly, stopper, and place in the cooling bath for fifteen minutes. Prepare a standard for comparison at the same time and in the same manner using 2 cc of the standard citral solution. Remove and compare the colors developed. Calculate the amount of citral present and repeat

the determination using a quantity sufficient to give the sample approximately the strength of the standard. From this result calculate the amount of citral in the sample. If the comparisons are made in Nessler tubes, standards containing 1, 1.5, 2, 2.5, 3, 3.5, and 4 mg should be prepared and the trial comparison made against these, the final comparison being made with standards between 1.5 and 2.5 mg varying but one-fourth of a milligram.

The following points are to be especially noted:

The aldehyde-free alcohol (25 cc) on standing for 20 minutes in the cooling bath with the fuchsin solution (20 cc) should develop only a faint pink coloration. If a stronger color is developed, treat again with metaphenylene-diamin hydrochlorid.

It is absolutely essential to keep the reagents and comparison tubes at the required temperature. Comparisons should be made within one minute after removing the tubes from the bath. Where the comparisons are made in the bath (this is possible only where the bath is glass) the standards should be discarded within twenty-five minutes after adding the fuchsin solution. Give samples and standards identical treatment.

Note on samples colored with turmeric whether or not the color interferes with the comparisons. On samples 2 and 3, after making determinations on the samples sent, repeat them, removing the colors as follows: After weighing the sample to be used for analysis in a glass stoppered weighing bottle, add a drop of concentrated hydrochloric acid and a small piece of fat-free woolen cloth, stopper and allow to stand over night. Remove the cloth washing with aldehyde-free alcohol and determine the citral in the colorless solution as usual. Repeat the above comparison heating the acidified sample and woolen cloth under a reflux cooler for a few minutes, cool, remove the cloth and determine the citral as usual.

The samples sent were as follows:

No. 1. A lemon extract containing 3,008 grams of 95 per cent alcohol and 192 grams lemon oil, the whole colored with turmeric.

No. 2. A terpenless extract of lemon strengthened with citral; 300 grams lemon oil dissolved in 1,796 grams of 95 per cent alcohol; 2,070 grams of water were added and after standing over night the precipitated oil was removed and 3.76 grams of citral added. The whole colored with Naphthol Yellow S.

No. 3. A solution of citral in dilute alcohol (50 per cent volume) containing 3,000 grams alcohol, 3.6 grams c. p. citral making the actual percentage of citral 0.12 per cent. The whole colored with Naphthol Yellow S.

No. 4. A solution of citral in dilute alcohol (50 per cent by volume) containing 3,500 grams alcohol and 2.12 grams c. p. citral making the actual percentage of the latter 0.061. The whole colored with turmeric.

The results reported are given in the following table:

Collaborative work on determining citral in lemon extracts, 1908.

Collaborator.	Without removal of color.				After removal of color.			
					By heating.		At ordinary temperature.	
	No. 1.	No. 2.	No. 3.	No. 4.	No. 2.	No. 3.	No. 2.	No. 3.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
R. W. Hiltz, Philadelphia, Pa.....	0.330	0.330	0.125	0.060			0.28	0.108
A. W. Hansen, New York.....	.322	a. 407	.136	.067		0.110	.386	.136
W. L. Dubois, Buffalo, N. Y.....	.328	.329	.107	.056	0.140	.089	.233	.100
A. P. Sy, Buffalo, N. Y.....	.318	.317	.116	.056	.110	.090	.278	.102
A. L. Cook, San Francisco, Cal.....	.286	.315	.118	.054	.228	.085	.188	.079
L. A. Brown, Agricultural College, North Dakota.....	.288	a. 370	a. 190	a. 100				
F. D. Merrill, Detroit, Mich.....	.354	.324	.133	.064	.202	.031	.333	.080
R. S. Hiltner, Denver, Colo.....	.326	.316	.137	.084	.215	.137	.311	.138
C. O. Dodge, Washington, D. C.....	.308	.312	.133	.060			.294	.116
S. H. Baer, St. Louis, Mo. a.....	a. 340	a. 387	a. 165	a. 140				
A. E. Taylor, Savannah, Ga.....	.360	.340	.125	.070	.290	.110	.290	.110
I. W. Brandel, Seattle, Wash.....	a. 143	a. 385	.109	.050				
Averages.....	.322	.323	.124	.062				
Maximum above average.....	.038	.017	.013	.022				
Maximum below average.....	.046	.011	.017	.017				
Theoretical amount.....			.12	.061				

a Excluded from averages.

COMMENTS OF ANALYSTS.

R. W. Hills: The methods submitted for this work were adhered to with the exception that in the removal of color from samples 2 and 3 the portions were weighed out into the 50 cc graduated, glass-stoppered flask, acidified as directed, and the piece of fat-free woolen cloth added (about 1.5 inches square). After standing over night the volume was completed with aldehyde-free alcohol, *without* removing the cloth.

Preliminary tests of the samples were made against a series of standards, but all final determinations were made by matching in the colorimeter. Final comparisons were always arranged so that the depths of tints compared were within 10 per cent, generally less, of equal strength.

Results reported are calculated from averages of four to five readings made in rapid succession with columns of 40 mm and 30 mm, i. e., 8 to 10 readings. Comparisons on the different depths of liquid gave concordant results.

Color in samples 1 (turmeric) and 2 (Naphthol Yellow S) gave no trouble whatever in comparisons. The samples are so highly diluted in the final determination that the color does not interfere. On sample 3 (Naphthol Yellow S) considerably more of the original liquid is present in the comparison tube, due to its lower citral content, and a very slight modification of tint in depths of 40 mm was noticed. With depths of 30 mm there was no apparent difference and tints were matched with ease. Sample 4 (turmeric) behaved similarly to No. 3. In depths of 40 mm there was a slight difference of tint, because nearly 3.5 cc of the original liquid was present in the tube. This slight difficulty disappeared in depths of 30 mm. Samples 2 and 3 were very satisfactorily decolorized by the treatment with the cloth. However, in so far as ease of comparison is concerned this treatment seems superfluous if comparisons are made with comparatively short columns of liquid, as above noted.

A. W. Hansen: The operator could not see that the color interfered with the comparisons.

W. L. Dubois: The comparisons were made in wide Nessler tubes graduated to 100 cc which were cooled to 15° in a large bath and for comparison placed in a tall beaker containing water at 15° and around which was wrapped a piece of white paper, the beaker being set on a white surface and lifted therefrom a few inches at the time of reading. The color in samples 2 and 4 did not seem to interfere with the determinations. The fuchsin sulphite solution when made as directed retained a slightly brownish tint. The fuchsin, however, which we had available for the preparation of this solution was not labeled c. p. and this possibly may have accounted for our failure to get a perfectly colorless solution.

C. L. Cook: None of the readings of any of the samples was interfered with by the presence of the coloring matter used. It was found necessary to allow the fuchsin solution to stand at least forty hours before a blank could be obtained with the aldehyde-free alcohol we were able to distil.

F. D. Merrill: Samples 1 and 4 colored with turmeric gave a color differing somewhat from the standard used in the determination of citral. In Nos. 2 and 3 colored with Naphthol Yellow S less difficulty was experienced in matching colors with the standards in the determination of citral when the original extract was used, but when the sample was decolorized by either method suggested it had a very different color as compared with the standard used in citral determination, and great difficulty was experienced in matching colors.

R. S. Hiltner: The small amount of turmeric in samples No. 1 and No. 4 did not interfere perceptibly with the color comparisons.

Sample No. 2, when heated with hydrochloric acid and woolen cloth under reflux condenser as directed, turned brown, apparently due to decomposition of citral. A somewhat similar change took place with No. 3, but to a less degree.

The same result was obtained on these two samples by simply acidifying with hydrochloric acid and treating at once with fuchsin reagent as by allowing the acidified solution to stand over night in contact with wool.

I was unable to secure alcohol that would not respond to the fuchsin test for aldehyde, even after prolonged standing and heating with m-phenylene diamine hydrochloride.

Besides the figures obtained by the trial method, Mr. Hiltner, of the Denver Food Inspection Laboratory, submitted a set obtained by a method devised by himself using metaphenylene diamine as a substitute for the fuchsin sulphite reagent. The writer makes the following claims for the method:

First. Since there is no color reaction with acetaldehyde, more correct results may be secured in the analysis of commercial extracts.

In the preparation of these extracts, ordinary rectified alcohol is, of course, used. Such alcohol always contains more or less acetaldehyde. Any general reagent for aldehydes, like fuchsin, therefore tends to give too high results for citral because of the reaction on the acetaldehyde present.

Second. It is unnecessary, as stated, to use especially purified alcohol free from aldehydes.

Third. All the operations may be carried on at room temperature.

The following figures were submitted on the official samples: No. 1, 0.251; No. 2, 0.305; No. 3, 0.117; No. 4, 0.061.

Nos. 1 and 2 are somewhat below the average figures submitted by the collaborators. Nos. 3 and 4 are much closer to the actual amount present than those obtained by Mr. Hiltner with the method under trial. As the method was called to the referee's attention only a few days before the meeting, no opportunity was offered to test it this year.

GENERAL DISCUSSION OF RESULTS.

The results obtained on the official samples as a whole exceed greatly the expectations of the referee.

When twelve different analysts are working even with a well-established method under varying conditions, experience has shown that some discordant results are apt to be obtained. When like discrepancies have been obtained with the official methods for nitrogen and potash, it would seem that the results, in the present case, are highly satisfactory.

It appears to be of no advantage to remove the color before making the determinations; in fact, several of the collaborators are of the opinion that it renders the solutions harder to read. The work done at Washington also indicated that there was little advantage to be obtained, certainly not sufficient to offset the loss of citral. The results were slightly better on the alcoholic solutions of citral than upon the extracts. They were better on the terpeneless extract than on the extract containing lemon oil. This is, in all probability, due to the effect of the non-aldehydic constituents upon the color of the fuchsin solution. Where the colors are not of like tint, considerable experience is required in order to correctly match them.

On the final comparisons the standard and sample must contain approximately equal amounts of citral; a deviation of over 10 per cent is not allowable.

The method is not difficult of manipulation, but does require pure reagents, especially in the case of aldehyde-free alcohol. It is highly probable that the greater part of the discordant results are due to the latter. Given a cologne spirit of good quality, there seems, however, to be no reason why good results should not be obtained. It is recommended that the method as submitted for the determination of citral in lemon extracts be adopted provisionally by the association.

REPORT ON SPICES.

By A. L. WINTON, *Associate Referee*.

The attention of the associate referee was directed to the adulteration of paprika with olive oil, and the methods of detecting this form of adulteration, by papers presented by Doolittle and Ogden and by Loewenstein at the New Haven meeting of the American Chemical Society. Although the time was short for giving this matter suitable attention, a circular letter was sent out on September 5 to such chemists as had previously expressed a willingness to cooperate, and later, samples of two kinds of paprika were distributed, one purporting to be pure, the other adulterated with olive oil.

The methods submitted for study are as follows:

METHODS.

NON-VOLATILE ETHER EXTRACT.

Dry in a desiccator over night or until the moisture is largely removed a sufficient amount of the material to yield an extract of from 0.2 to 0.25 grams. Extract according to the official method for the determination of crude fat (Bul. 107, Rev., p. 39, 5 (b) (1)), collecting the ether solution in a tared flask. Dry the extract at 100° C. for 15-minute periods until constant weight is secured.

IODIN NUMBER.

Determine by the Hanus method (Bul. 107, Rev., pp. 136-7), using the extract obtained as described in the preceding section.

Great care should be exercised in weighing the flask, both before and after extraction, as an error of 1 milligram is equivalent to an error of over 0.5 in the iodine number. A glass-stoppered 200 cc Erlenmeyer flask may be used for the extraction and also, without transfer, for the determination of the iodine number, although in our experience more accurate results may be secured by using a vial-mouth unstoppered flask of about 40 cc capacity, thus reducing the exposed surface to a minimum. In the latter case the flask, after dissolving the extract in chloroform and adding the Hanus solution, is introduced into a saltmouth, glass-stoppered bottle, broken with a glass rod and the titration carried out in this bottle in the usual manner.

It was suggested that in extracting the fat 3 grams of the pure paprika and 2 grams of the paprika adulterated with oil be used, thus securing amounts of extract suitable for determination of iodine number.

ALCOHOL EXTRACT.

Follow the official method (Bul. 107, Rev., p. 163).

DISCUSSION OF RESULTS.

The results obtained by the five analysts who took part in the cooperative work are given in the following table:

Analysis of pure paprika and samples mixed with olive oil.

Collaborator.	Pure paprika.			Paprika with olive oil.		
	Alcoholic extract.	Non-volatile ether extract.	Iodin number.	Alcoholic extract.	Non-volatile ether extract.	Iodin number.
	Per cent.	Per cent.		Per cent.	Per cent.	
Genevieve Imus, Minnesota.....	13.36		76.5	14.96		97.4
	13.33		76.2	14.97		96.7
C. D. Woods, Maine.....		5.43	82.0		15.30	76.1
		5.81	77.4		13.18	80.7
C. P. Wilson, Washington, D. C.....	10.44	6.10	105.9	13.43	13.76	106.4
C. S. Brinton, Philadelphia.....	12.8	5.27	105.8	13.4	12.01	
	12.7	5.36	105.2	13.35	12.10	^a 115.05
C. I. Lott, Chicago.....	A 10.62	5.52	114.6	12.92	12.58	115.2
		5.54	112.3		12.64	108.0
		5.37	116.7		12.72	112.0
	B 10.50	5.45	117.2		12.71	116.1
		5.47	112.9	12.76	12.52	107.2
		5.32	117.7		12.42	115.6
	C 10.75	5.47	115.2		12.51	111.4
		5.46	113.1	12.96	12.80	105.1
		5.40	115.1		12.75	105.8
					12.79	116.1
					12.60	117.6

^a Average.

Genevieve Imus: This collaborator states that through a misunderstanding the portions taken for analysis were weighed out after drying the materials in a desiccator. For this reason the percentages of alcohol extract and nonvolatile ether extract are not comparable with those given by the other analysts and are not given in the table.

C. D. Woods: The ether extract in the determinations made by the method described not being complete, other trials were made, using different quantities of the material and extracting for longer periods. The results are given in the following table:

Analyses of pure paprika and samples adulterated with olive oil, varying weight of sample, and time of extraction (Woods).

PURE PAPRIKA.

Weight used.	Time of extraction.	Non-volatile ether extract.	Iodin number of non-volatile ether extract.
<i>Grams.</i>	<i>Hours.</i>	<i>Per cent.</i>	
3	16	5.43	82.0
3	16	5.81	77.4
3	24	5.48	87.3
3	24	5.74	79.3
3	24	5.91	77.9
1	100	13.15	-----
1	100	13.93	42.6

PAPRIKA ADULTERATED WITH OLIVE OIL

2	16	15.30	76.1
2	16	13.18	80.7
1	24	12.23	93.5
1	24	13.65	84.0
1	24	13.85	84.1
1	100	18.37	66.2
1	100	18.70	65.8

Mr. Woods comments on the above results as follows:

I note that Doolittle and Ogden report a much higher iodine number than any of my results would indicate and also that their results are very concordant. With directions as given I fail to see how one could place any reliance on the results of this determination. It may be possible that by running the ether extraction for an exact definite time results can be obtained agreeing reasonably close with each other, but I doubt this somewhat, for it has been our experience that some determinations extract much faster than others, depending on the rate of flow of the ether and the type of extractor used, and that until the extraction is complete there is no surety that two determinations will agree at any given time during the process.

These few determinations seem to indicate that if the iodine number is made on the complete ether extract other material besides fat (resins, etc.) will so increase the weight that the value of the iodine number will be reduced, while, if the determination is made before the extraction is complete, the results can not be depended on to agree.

C. S. Brinton comments on his results as follows:

The iodine numbers on the nonvolatile ether of the samples prepared with oil did not agree, and I am reporting only the average of results obtained. I was very much surprised to find the iodine number of the nonvolatile ether extract in the pure sample so much lower by the method you suggest than that obtained by the method used by Doolittle, but this is easily accounted for, because a long extraction with ether carries other material which is not readily soluble in ether and would not be found in the ether extract when a shorter extraction time is used. From the results obtained by this method I do not think that it would be advisable to use an official ether extract for the determination of the iodine number, as by so doing we are liable to overlook samples prepared with olive oil, the presence of which would be revealed by using Doolittle's method.

C. P. Wilson stated that he was not entirely satisfied with the results because with the apparatus he used he found it necessary to dissolve the fat before removing it from the flask in which it was recovered by the extraction.

C. I. Lott: In order to secure evidence with regard to the accuracy of the sampling, analyses were made of three bottles (A, B, and C) of each paprika. The discrepancies in the determination of the iodine number were attributed partly to differences in the amount of extract obtained occasioned by the removal of different amounts of the difficultly soluble resins and partly to errors in the process of determining the iodine number. It was suggested that possibly in the earlier determinations the extract was not completely dissolved in the chloroform preliminary to the Hanus solution. In the later determinations special effort was made to secure a complete solution. With this precaution the following results were obtained: Pure paprika, 116.7, 117.7, 115.1; paprika with olive oil, 116.1, 116.1, 117.6. Further experiments are needed to ascertain whether or not a better agreement of results can be secured by observing special precautions in dissolving the extract.

CONCLUSIONS.

The radical difference in the results reported by the different analysts in the determination of nonvolatile ether extract and the iodine number of the extract may be in part explained by differences in the extraction apparatus employed and in the rate of extraction, some of the analysts securing an extract which contained a much greater amount of resins than that obtained by the others, which resins have a much lower iodine number than the fatty oil. This explanation, however, does not account for many of the differences. For example, Messrs. Woods and Lott obtained practically the same percentages of nonvolatile ether extract in the pure paprika, but one reports an average iodine number of about 80 and the other of about 115. On the other hand, Mr. Wilson obtained the highest percentage of nonvolatile ether extract, and Mr. Brinton the lowest, yet both secured practically the same results on the iodine number.

The results reported indicate either that the method of securing the nonvolatile ether extract for the determination of iodine number is seriously at fault, or else special precautions, yet to be determined, are necessary to the success of the process. The results are not only widely discrepant, but they fail to throw any light whatever on the question of adulteration.

RECOMMENDATIONS.

It is suggested that during the ensuing year the following methods be studied: First, extraction on filter paper, with ether, as followed by Doolittle and Ogden,^a and, second, shaking for a definite time with a definite volume of ether and evaporation of a portion of the filtered extract. It is believed that satisfactory results can be obtained only by a purely conventional method, using the same weight of material, the same volume of ether, and the same time of extraction. It may be found important, however, to use such portions of the ether solutions as will yield in all cases approximately the same amount of nonvolatile ether extract. The results obtained in the determination of alcohol extract throw no light on the question of added oil.

REPORT ON COLORS.

By H. M. LOOMIS, *Associate Referee*.

The work of the past year has been chiefly on the identification of colors. For this purpose twelve samples of colored food products were prepared in the laboratory, using the purest colors available, and samples of each were sent to six cooperating chemists. It is only just to state that many of the colors used were not furnished as food colors by the manufacturers. In this work the endeavor has been to prove that the colors used were simple commercial colors, and not mixtures, without special regard to the presence of mineral salts, etc.

Since the promulgation of F. I. D. 76 of the Board of Food and Drug Inspection, allowing the use of certain coal-tar colors in food products and prohibiting all others, it has become quite necessary for food chemists to make a study of the methods of identifying colors to find out with what degree of accuracy these methods serve their purpose. In making this study it is of course very essential to work with pure colors, and as the time available for this work would not allow of preparing these colors in the laboratory, there were used colors furnished by manufacturers, who in most cases gave both the commercial and the scientific name of these samples, and upon them such tests were made as seemed necessary to establish fully the fact that they corresponded with the names given and were simple unmixed colors.

No originality in the methods of testing is claimed, the standard works of reference on the subject having been freely consulted. In every case the well-known tests by color reactions in aqueous solution, on the dyed fiber, and with concentrated sulphuric acid on the dry color were used. This includes a test for mixed colors made by sprinkling dry color on a surface wet with water or concentrated sulphuric acid. In addition the following tests were made on the separate colors:

TARTRAZIN:

Precipitation by alcohol: Concentrated aqueous solution + 95 per cent alcohol = crystalline yellow precipitate.

0.1 per cent aqueous solution + stannous chlorid = yellow precipitate, soluble in oxalic acid solution (10 per cent).

0.1 per cent aqueous solution + barium chlorid solution = yellow precipitate.

0.1 per cent aqueous solution + calcium chlorid solution = no precipitate.

Conclusion: Pure color, S. & J. 94.

SAFFRON:

Test for coal-tar colors; microscopic examination.

Conclusion: Pure color.

NAPHTHOL YELLOW S:

Color reaction with stannous chlorid and ammonia. Test for organic sulphur.

Deflagration test: Heated on platinum foil. Takes fire explosively.

Solubility in ether: Nearly insoluble in neutral or acid solution.

0.1 per cent aqueous solution + barium chlorid solution (10 per cent) = orange precipitate, insoluble in acetic acid.

0.1 per cent aqueous solution + calcium chlorid solution (10 per cent) = no precipitate.

0.1 aqueous solution + lead acetate = orange precipitate, soluble in acetic acid.

0.1 per cent aqueous solution + cobalt chlorid and caustic soda = olive-green precipitate.

Remarks: Color nearly pure. Contains small amount of unsulphonated naphthol yellow.

TROPAEOLIN O O:

Reduction of color by stannous chlorid in acid solution; separation of para aminodiphenylamin from alkaline solution by ether. Melting point, 61° to 62° C.

Precipitation by salt: 0.1 per cent solution of color + few drops 10 per cent sodium chlorid solution = precipitate of color.

0.1 per cent aqueous solution + barium chlorid (10 per cent) = colored precipitate, like $\text{Fe}(\text{OH})_3$.

0.1 per cent aqueous solution + calcium chlorid (10 per cent) = colored precipitate, like $\text{Fe}(\text{OH})_3$.

Conclusion: Pure color, S. & J. 88.

ERYTHROSIN: (Color used in sample 3 C.)

Aqueous solution, pink fluorescent (shows presence of other colors besides erythrosin.)

Color extracted from acidified aqueous solution by ether. Ether solution washed several times, evaporated and color dried.

Halogens: Chlorin, bromin, and iodine found in color qualitatively. (Mulliken "Identification of Organic Compounds," p. 13). Determination of bromine and iodine by Janasch and Aschoff method gave 17 per cent bromine and 9.7 per cent iodine.

Color a mixture of eosin colors containing chlorin, bromine, and iodine.

RHODAMIN:

Contains no bromine or iodine; 0.4 per cent ash; insoluble, even on boiling in caustic potash solution; sp. gr. 1.3.

Aqueous solution pink; yellow fluorescence, which disappears on warming and reappears on cooling.

0.01 per cent aqueous solution + stannous chlorid solution = bright crimson precipitate, purplish by transmitted light.

0.01 per cent aqueous solution + tannin reagent; test for basic color = precipitate.

Benedikt's test with zinc and ammonia: (Allen, "Commercial Organic Analysis," vol. 3, part 1, page 322.)

Conclusion: Pure color, S. & J. 504.

ROSE BENGAL:

Qualitative analysis shows halogens, iodine, and chlorine—no bromine.

Benedikt's test with zinc dust and ammonia. (See Allen, loc. cit.)

Benedikt's test: Boiling with caustic potash solution. (Sp. gr. 1.3.) (See Allen.)

Color separated from acidified aqueous solution as in the case of erythrosin.

Quantitative determination of iodine and chlorine.

Chlorine determined by silver nitrate after removal of iodine by nitrous acid and carbon bisulphide.

Iodine determined from total halogens by difference.

Chlorine, 8.89 per cent; iodine, 49.5 per cent; ratio = 1 to 5.56.

Ratio of halogens in tetraiodo-dichloro-fluorescein = 1 to 7.2.

Ratio of halogens in tetraiodo-tetrachloro-fluorescein = 1 to 3.6.

Conclusion: Color a mixture of the two Rose Bengals, S. & J. 520 and 523.

PHLOXIN:

Qualitative analysis shows presence of halogens, bromine, and chlorine; no iodine.

Determination of bromine by Mohr's method gave 39.8 per cent in color, purified by extraction with ether.

Chlorine, from total halogens by difference = 11.9 per cent.

Ratio — chlorine : bromine = 1 : 3.34.

Benedikt's test with zinc dust and ammonia. (See Allen.)

Benedikt's test with boiling potassium hydroxide solution. (See Allen.)

Conclusion: This color is a mixture of the two phloxins, S. & J. 518 and 521.

COCHINEAL RED A, S. & J. 106:

Tested for mixed color by precipitating part of color from solution with salt, filtering and dyeing wool to same depth with filtrate and solution of precipitated color. Both dyeings were nearly the same shade, indicating fairly pure color.

Dry color sprinkled on concentrated sulphuric acid shows small amount of foreign color.

Reduction with stannous chloride and hydrochloric acid, making alkaline with sodium hydroxide and extracting, gave very little ether-soluble matter. This shows absence of colors yielding ether-soluble bases on reduction.

Conclusion: Color is fairly pure, but contains a small amount of foreign color.

FAST RED C, S. & J. 103:

Tested for mixed color, as in the case of cochineal red A, by fractional precipitation with salt and by sprinkling on concentrated sulphuric acid. Small amount of foreign color shown.

Color is fairly pure, but contains a small amount of foreign color.

PONCEAU 2R or 3R:

0.1 per cent aqueous solution + barium chloride solution (10 per cent) = crimson precipitate, insoluble in acetic acid.

0.1 per cent aqueous solution + calcium chloride solution (10 per cent) = no precipitate.

0.1 per cent aqueous solution + lead acetate solution (10 per cent) = crimson precipitate.

Color reduced with stannous chloride and hydrochloric acid. Solution made alkaline with caustic soda and distilled with steam. Liquid amido compound distills over, which could not be solidified in ice water. Boiling point about 215° C. This shows the amido compound to be xylidine.

Conclusion: Color is ponceau 2R or xylidine red, S. & J. 55.

ACID GREEN:

Solubility in absolute and 95 per cent alcohol; no sign of mixed color by sprinkling on wet filter; no chlorine in the ash.

Conclusion: Pure color, S. & J. 435.

PERSIAN BERRY EXTRACT:

Reactions correspond very closely to those of a buckthorn berry extract prepared in the laboratory.

The accompanying table shows the results obtained in the identification of the colors. Considering the fact that three of the collaborators had never undertaken work on the identification of colors before, the results appear to be quite satisfactory with regard to the coal-tar colors.

Results of cooperative work on the identification of colors.

No.	Material colored.	Color used.	Reports of analysts.				
			C. S. Brinton.	Hare, Mitchell, and Pringle.	F. O. Woodruff.	Hayward and Allen.	E. J. Shanley.
1 F	Lemon extract.....	Tartrazin (Bad.) S. & J. 94.	Tartrazin. S. & J. 94.	Wood yellow T. (Tartrazin)	Tartrazin.....	Not reported.	Tartrazin. S. & J. 94.
2 F	Lemon extract.....	Saffron.....	Saffron.....	Chrysoidin	Vegetable color.....	do.....	Saffron.
3 F	Lemon extract.....	Naphthol yellow S. & J. 4.	Naphthol yellow S. & J. 4.	Yellow Y. M. (R. H.) (Naphthol yellow S.)	Naphthol yellow S.....	do.....	Naphthol yellow. S. & J. 4.
4 F	Lemon extract.....	Tropaeolin O.O. (Cassia S. & J. 88.)	Tropaeolin O.O. (Possibly metanil yellow)	Orange IV. (Tropaeolin O.O.) S. & J. 88.	Metanil yellow (S. & J. 88.)	do.....	Orange IV. (Tropaeolin O.O.) S. & J. 88.
1 C	Cherries.....	Cochineal Red A. (Bad.) S. & J. 106.	Not reported.	Cochineal Red A.....	Ponceau 6R. S. & J. 108.	do.....	Scarlet 6R. S. & J. 108.
3 C	Cherries.....	Mixture of S. & J. 50-52.	Eosin A. S. & J. 512.	Eosin A. S. & J. 512.	Eosin.....	do.....	Eosin. S. & J. 512.
4 C	Cherries.....	Rose Bengal (Bad.) S. & J. 55.	Rose Bengal. S. & J. 520.	Rose Bengal.....	(?)	do.....	Rose Bengal.
V	Pumpkin pulp.....	Ponceau 2R (Sch.) S. & J. 55.	Ponceau 3R. S. & J. 56.	Ponceau 3R. S. & J. 56.	Xyldin Red, Ponceau 2R. S. & J. 55.	Ponceau 2R or 3R.....	Cochineal Red A. S. & J. 106.
VI	Sugar sirup.....	Buckthorn (Persian berry extract)	Similar to Weld.	Alizarin yellow A?	Caramel.....	Not reported.	Fustic.
VIII	Sugar sirup with caramel.....	Phloxin (Bad.) S. & J. 518 & 521.	Phloxin. S. & J. 521.	Phloxin.....	Phloxin.....	do.....	Eosin A. S. & J. 512.
IX	Sugar sirup with caramel.....	Fast Red C (Bad.) S. & J. 103.	Not reported.	Bismarck brown.....	Fast red C.....	do.....	Fast Red D. (S. & J. 107.)
X	Sugar sirup.....	Acid green (Cassella.) S. & J. 435.	Light green S. F. yellowish. (Acid green.) S. & J. 435.	Acid green O.O. (Sch.) S. & J. 435.	Acid green.....	Acid green.....	Acid green. S. & J. 435.

NOTES AND COMMENTS BY THE COLLABORATORS.

C. S. Brinton used the tables of Rota and others given by Allen,^a Schultz and Julius, "Organic coloring matters," and Circulars 25 and 35, Bureau of Chemistry. Considerable difficulty was encountered in some cases in isolating color from fruit pulp and sirup. Double-dyeing method was used for extracting color from material, and color was obtained in aqueous solution by extracting wool with ammonia. Sample VI gave considerable trouble, and definite report was not made.

F. O. Woodruff used chiefly tables of Green, Yoeman, and Jones,^b also tables in Allen and in Schultz and Julius, and Circular 35, Bureau of Chemistry. He says: "Three difficulties attending identification are: (1) A commercial dye from different manufacturers varies in purity and therefore in properties, though bearing the same or a synonymous trade name; (2) amount of color on dyed fiber or in color solutions affects the nature of the reactions therewith; (3) ordinary description of color reactions varies with the observer and does not allow of fine distinctions."

Hare, Mitchell, and Pringle used Rota's table and those in Circular 35, Bureau of Chemistry. They comment as follows: "We find Rota's scheme quite valuable in assisting us in the general classification of the dye. An accurate and complete color chart would be a great aid, especially to those not used to making sharp color distinctions."

E. J. Shanley used the tables in Allen and Circular 35, Bureau of Chemistry.

RECOMMENDATIONS.

It is recommended—

(1) That an effort be made to obtain authentic samples of vegetable or natural coloring matters, such as are used in food products. This work should be assigned to such men as are in a position to obtain authentic samples, for it is well-nigh impossible for one person to obtain any considerable number of such samples and to ascertain their source and method of preparation;

(2) That characteristics of vegetable coloring matters and methods for identification be studied;

(3) That synthetic preparations of pure colors for standards be made;

(4) That the separation and identification of mixed colors be studied.

The president announced the following appointments as members of Committee A on recommendations of referees: R. J. Davidson, J. P. Street, J. G. Lipman, B. L. Hartwell, and W. A. Withers.

The association adjourned until 2 o'clock.

THURSDAY—AFTERNOON SESSION.

REPORT ON MEAT AND FISH.

By F. C. WEBER, *Associate Referee*.

In view of the fact that no work has ever been reported to the association on this subject, it seemed to the referee that some results showing the degree of accuracy of some of the chemical methods ordinarily employed in separating protein nitrogen, and at what point they show deterioration of meats, might be of interest. Owing to the nature of the work and the difficulty of keeping samples uniform, no attempt was made to secure collaborative work.

SAMPLES.

The determinations here reported were made on three samples of chicken meat. Six young market chickens were obtained, killed, dressed, and allowed to stand in the ice box over night. The next morning the flesh was separated from the bones

^a Commercial Organic Analysis, vol. 3, part 1.

^b Soc. Dyers and Colorists, 1905, 21: 236. Digitized by Google

and skin and thoroughly ground and mixed by passing six times through a meat chopper. It was then divided into two equal portions, one marked "fresh" and the other, after the addition of 0.1 per cent of boric acid, was marked "preserved."

The third sample represents the meat from three cold-storage drawn chickens, in storage twenty-six months, treated in the same manner as above, but without the addition of boric acid, and marked "stored." Each sample was placed in a screw-cap Mason jar and allowed to stand for one week, at laboratory temperature during the day, and in an ice box at night. During this time samples were taken for analysis on the first, second, third, sixth, and seventh days of standing. Every precaution was observed to guard against loss of moisture during the removal of the sample, as a result of which the moisture content remained very constant.

METHODS.

The following determinations were made at each of the periods cited: Moisture, total nitrogen, ammonia nitrogen, and, in the aqueous extract at room temperature and with ice water, nitrogen was determined as total, coagulable, amido, and ammonia. The difference between the sum of the coagulable and amido nitrogen and the total soluble nitrogen is considered as proteoses and peptones. The fat was determined once at the beginning of the experiment.

Moisture was determined on a 2-gram sample, dried in a water oven for ten hours. The loss of weight was calculated as moisture.

Fat.—The dried sample from the moisture determination was ground with dry sand and extracted with anhydrous ether in a Knorr extractor for twenty-four hours for the determination of fat.

Nitrogen determinations were made in the Nitrogen Section of the Bureau of Chemistry by Mr. H. W. Houghton, using the Gunning modification of the Kjeldahl method.

The *ammonia nitrogen* was determined on from 5 to 10 grams of sample distilled from a 750 cc flask, after the addition of 250–300 cc water and 10 grams magnesium oxid. The distillate was collected in standard acid and the ammonia nitrogen determined after a one-half hour distilling, 150 cc being distilled off. The distillation was continued for three half-hour periods, 150 cc of water being returned to the flask between each distillation. The results reported represent the sum of the three half-hour periods.

Water-soluble nitrogen [at room temperature (23°–25° C.) and with ice water (8° C.)]: Twenty grams of the well-mixed sample of meat were weighed into a 450 cc Erlenmeyer flask, 250 cc of water added, and shaken for three hours in a shaking machine. In the case of ice-water extract chopped ice was added from time to time, the volume in the flask being kept constant by decanting the excess of water into a second flask. After being shaken the required length of time, the flasks were placed in the refrigerator over night, a small quantity of thymol and phenol having been added as a preservative. The next day they were poured through linen bags and extracted with room temperature and ice water, respectively, by vigorous manipulation with the hands and successive portions of water, till the final extract gave a negative biuret reaction. The extraction was very tedious and required, at first, an entire day for completion, using from 2,200 to 2,500 cc of room-temperature water, and from 1,800 to 2,000 cc of ice water. The room-temperature extract was made up to a volume of 2,500 cc, while the ice-water extract was made up to 2,000 cc throughout the experiment, though the latter extractions, particularly on the last two days, were completed with from 1,400 to 1,800 cc water. After making to volume and thoroughly mixing, the solutions were filtered through 6-inch funnels containing a 38.5 cm S. & S. 588 folded filter paper. The first 750 cc which ran through was discarded (in the case of the room-temperature extract this was used for the ammonia determination); the second quantity, 600 cc to 800 cc, was used for the water-soluble nitrogen determinations.

The filtration of the solutions of the first three extractions was very simple, the solutions running through the paper readily, though the second portion was still somewhat cloudy. As the samples spoiled, the extraction became more easy and the filtration more difficult, until on the last two days it was quite difficult to obtain sufficient solution to make the determinations. This filtered extract was entirely clear. The total nitrogen in the aqueous extract was made on 100 cc of the solution.

Ammonia nitrogen was determined on 500 cc of the room temperature extract, by distillation with magnesium oxid.

The *coagulable protein nitrogen* was determined in a sample of 200 cc of the water extract. This was placed in a 300 cc evaporating dish and evaporated on the steam bath to a volume of 40 cc. The solution was neutralized with tenth-normal sodium hydroxid, using phenolphthalein as indicator, then replaced on the steam bath and allowed to evaporate for ten minutes, filtered on a plain filter, and washed with hot water. The filter and precipitate were transferred to a Kjeldahl flask and the nitrogen determined.

Amido nitrogen: The coagulable protein filtrate was made up to 100 cc volume and 50 cc employed for the amido nitrogen determination. The 50 cc were placed in a 100 cc graduated flask, 15 grams of sodium chlorid added, and the flask well shaken and placed in an ice box. A 24 per cent solution of tannin was prepared, filtered, and placed in the ice box. After one hour 30 cc of the 24 per cent tannin solution were added to each flask and the two flasks filled to the mark with ice cold water. The flasks were thoroughly shaken and stood in the ice box over night. A blank must be carried out simultaneously, as the best tannin contains some nitrogen. The solutions are filtered into 50 cc flasks and the nitrogen determined in the 50 cc. The nitrogen figure thus obtained multiplied by two, minus the nitrogen of the blank, gives the amido nitrogen in 50 cc of the coagulable filtrate.

The sum of the amido and coagulable nitrogen subtracted from the total soluble nitrogen is considered as proteoses and peptones. No effort was made to separate the albumoses, proteoses, and peptones. All the results are calculated to a moisture and fat-free basis and are also expressed in per cent of the total nitrogen of each day's analysis.

The ice water extractions were made by Mr. H. L. Amoss and the coagulable and amido nitrogen separations by Mr. F. C. Cook, both of the Bureau of Chemistry.

The methods as selected, while not representing all that might have been employed, were those that have been generally used in the Bureau of Chemistry, and it is hoped that the work may be used as a starting point in this subject and serve to show the accuracy of the methods when applied to meats in a progressive state of deterioration.

DISCUSSION OF RESULTS.

The moisture results show very little change throughout the period, the average in the case of the fresh and preserved samples being 73.00 and 71.70 per cent for the storage sample. There was 4.12 per cent of fat in the fresh chicken and 4.09 per cent in the storage. The results on total nitrogen (see table, page 48) are as uniform throughout as the nature of the material and the accuracy of sampling would permit, and serve to show that there is no gaseous loss of nitrogen, while the ammonia nitrogen (that determined directly on the sample, as well as that determined in the extract) is markedly increased throughout and very uniform, particularly in the case of the stored and preserved samples. The amount is quite small at the time of the first analysis and remains so till the third analysis (made after standing two days), when the storage sample contains a little more than the other samples. From this point the increase is rapid. The variations in percentage amounts are from practically 1 per cent in all cases on the first analysis, to 11, 15, and 13 per cent for the fresh stored and preserved samples, respectively, on the last analysis, after seven days standing. The ammonia results on the water extract were unfortunately not made on the first day. They show practically the same results as those determined directly, but are not quite so uniform and not so high in amount. In the case of the formation of ammonia, the increased amount seems to begin to be formed after two days standing.

In connection with these changes it may be well to state here the changes in the samples which could be observed macroscopically. At the time of first analysis the samples were fresh, the storage sample showing a characteristic dried appearance. After standing one day they were practically the same, though what may be termed a slight fermenting action seemed to be taking place. On standing two days the samples had begun to deteriorate, especially the fresh and stored sample, while the preserved sample appeared fairly fresh. After three days standing, the deterioration was more

marked. A slight odor of spoiled meat was noticeable, more markedly in the fresh and stored meat than in the preserved. After standing six days the odor was quite bad; the samples had lost their texture and there was no doubt that they had spoiled. No difference in their physical condition could be detected after standing seven days that was not noticeable after six days standing.

The nitrogen determined in the water-soluble material at room temperature shows the total nitrogen extracted to be largely increased during the experiment, the first decided increase showing in the samples after standing two days. The coagulable nitrogen shows but a slight tendency to increase, the most marked and uniform change being in the stored sample. The amido nitrogen is not very uniform and shows a tendency to decrease especially where the samples are in an advanced stage of putrefaction. The nitrogen here termed proteoses and peptones is markedly increased during the final days of the experiment, the storage sample again showing a more uniform change. The increase of ammonia nitrogen in the water extract conforms to that determined directly, but is not quite so large in amount.

The nitrogen in the ice water extract in the various forms separated shows the same general trend as does that of the room temperature extract, though the amounts extracted are usually not so large.

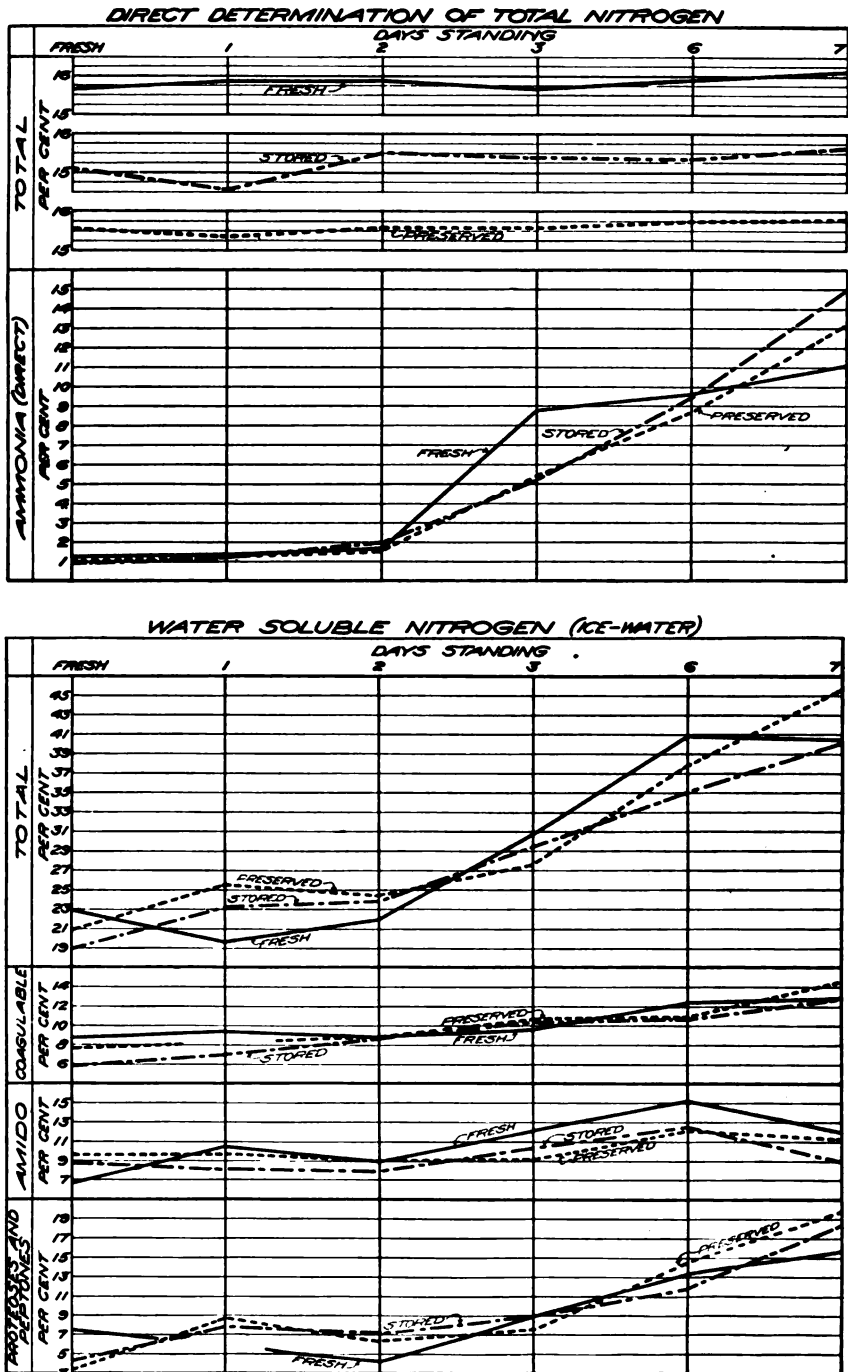
The graphic charts, figs. 3 and 4, show these changes more plainly. It is quite noticeable throughout that the results on the storage sample are very uniform and progressive and, moreover, in all but two instances, in all the determinations, the results on the first analysis show the storage sample to be lower in the various constituents than the fresh samples. The same general tendency seems to run throughout the experiment though one would expect the storage meat to deteriorate more rapidly.

Taking into consideration the variations in the determinations and the limitations of the methods themselves, there does not appear to be a very clearly defined point at which deterioration can be said to begin, unless it is shown by the ammonia and water-soluble total nitrogen determinations. The increase in these constituents coincides with the macroscopical observation and physical appearance of the sample. The use of ice water in the extraction is unnecessary, as the methods employed are not of sufficient accuracy to detect the greater changes from day to day in the early stages, much less any change which may be due to enzymic action during the process of extraction.

It seems probable from the results that the determination of ammonia may be a valuable asset in showing the first indications of changes, as these results are the most uniform and progressive. A large amount of work has recently been done on the methods for the determination of ammonia in animal and vegetable materials. Richardson ^a after considerable experimenting on the ammonia nitrogen determination, and in which he extracted the meat with 60 per cent alcohol and distilled with magnesium oxids, aspirating air through the flask, and distilling under reduced pressure, finally adopted the method as outlined above as best suited to the purpose.

His results on pure ammonium chlorid distilled in a vacuum with magnesium oxid and 60 per cent alcohol are nearly theoretical. This is in substance the method as now employed in the determination of ammonia in urine and might be adapted to this work.

^a J. Amer. Chem. Soc., 1908, 30: 1515.



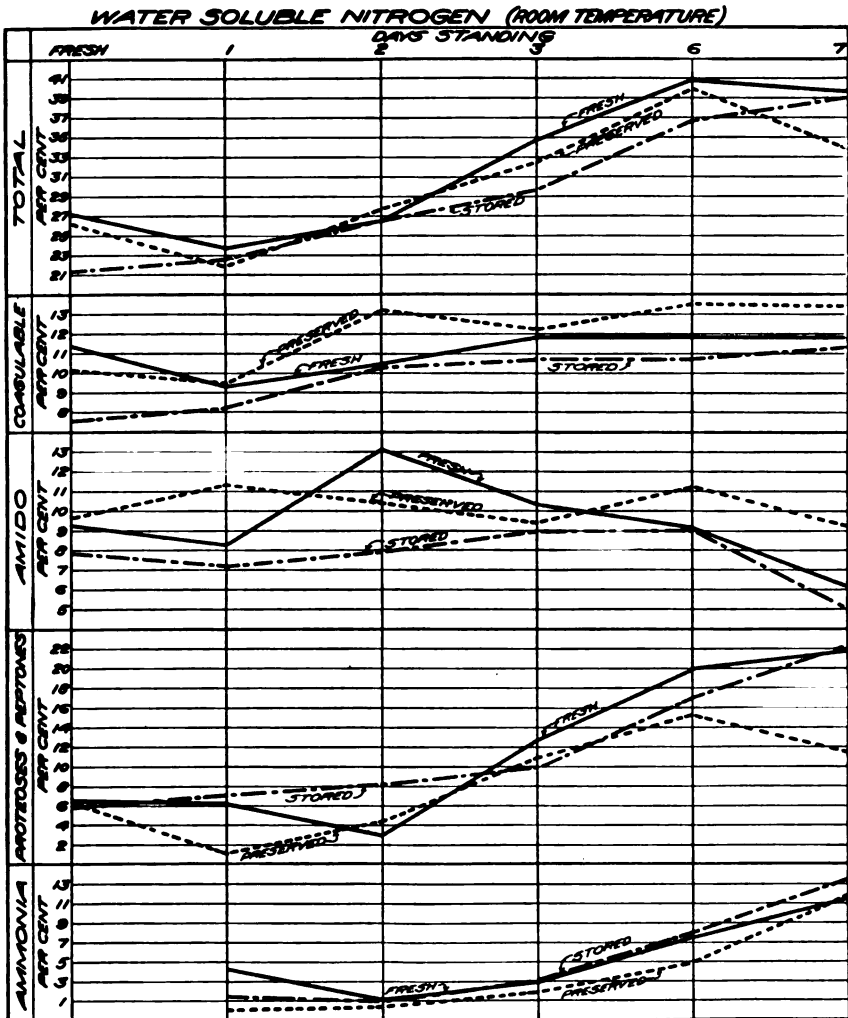


FIG. 4.—Changes taking place during seven days in the nitrogenous constituents (water-soluble at room temperature) of fresh, cold-stored, and preserved chicken meat.

Nitrogenous bodies in water extracts of fresh, stored, and preserved chicken meat.

[Water-free and fat-free basis. Black figures are results expressed in percentage of total nitrogen.]

Date.	Days elap.	Description.	Total nitrogen.			Ammonia nitrogen.			Nitrogen in water extract (room temperature) 22°-23° C., as—		
			Fresh.	Stored.	Preserved.	Fresh.	Stored.	Preserved.	Fresh.	Stored.	Preserved.
Total soluble.											
1908.			Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Oct. 13	0	Fresh; stored sample had characteristic dried appearance....	15.63	15.11	15.57	0.198	0.142	0.153	4.27	3.23	4.09
Oct. 14	1	All samples comparatively same; no signs of deterioration....	15.86	14.56	15.35	1.27	.94	.98	27.32	21.38	26.57
Oct. 15	2	Samples beginning to deteriorate; more marked in fresh and stored; preserved, fairly fresh.	15.90	15.51	15.63	1.33	1.18	1.26	3.76	3.29	3.36
Oct. 16	3	Slight odor noticeable, more marked in fresh and stored; samples were spoiling and becoming soft.	15.66	15.42	15.62	1.67	1.305	1.242	23.71	22.60	21.89
Oct. 19	6	Odor quite bad and samples spoiled; were soft and "mushy"....	15.93	15.37	15.75	8.81	5.19	5.40	4.21	4.11	4.34
Oct. 20	7	Same as on 19th.....	16.15	15.66	15.83	1.38	1.800	1.843	26.48	26.49	27.77
						1.53	1.46	1.37	5.44	4.56	5.06
						9.60	9.49	8.70	40.80	26.69	32.39
						1.81	2.35	2.09	6.41	6.11	5.37
						11.21	15.00	13.20	39.69	39.02	33.92

Date.		Days old.	Description.	Nitrogen in water extract (room temperature) 22°-23° C. as—													
				Coagulable.		Amido.		Proteoses and peptones.		Ammonia.							
				Fresh.	Stored.	Pre-served.	Fresh.	Stored.	Pre-served.	Fresh.	Stored.	Pre-served.					
1908.				Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	
Oct. 13	0		Fresh; stored sample had characteristic dried appearance.....	1.79	1.15	1.59	1.46	1.20	1.51	1.02	0.88	0.98	0.66	0.20	0	0	0
Oct. 14	1		All samples comparatively same; no signs of deterioration...	11.45	7.61	10.21	9.24	7.94	9.69	6.53	5.82	6.29	4.16	1.37	0	0	0
Oct. 15	2		Samples beginning to deteriorate; more marked in fresh and stored; preserved, fairly fresh.	9.33	8.31	9.45	8.26	7.21	11.24	6.05	7.07	1.11	1.16	1.12	1.2	1.2	1.2
Oct. 16	3		Slight odor noticeable, more marked in fresh and stored; samples were spoiling and becoming soft.	10.50	10.25	13.18	13.08	7.98	10.26	2.83	8.25	4.29	1.01	1.77	1.38	1.38	1.38
Oct. 19	6		Odor quite bad and samples spoiled; were soft and "mushy"....	11.81	10.70	12.23	10.28	8.96	9.25	12.64	9.86	10.82	2.94	3.06	1.79	1.79	1.79
Oct. 20	7		Same as on 19th.....	11.80	10.74	13.62	9.17	8.98	11.24	19.84	16.98	15.11	1.82	2.10	1.85	1.85	1.85
				11.83	11.62	12.39	6.13	4.98	9.16	21.61	22.41	11.37	11.27	12.41	11.69	11.69	11.69

Nitrogenous bodies in water extracts of fresh, stored, and preserved chicken meal—Continued.

Date.	Days old.	Description.	Nitrogen in water extract (boe water) 8° C., as—									
			Total soluble.			Coagulable.			Amido.		Proteoses and peptones.	
			Fresh.	Stored.	Pre-served.	Fresh.	Stored.	Pre-served.	Fresh.	Stored.	Fresh.	Pre-served.
1908.												
Oct. 13	0	Fresh; stored sample had characteristic dried appearance....	Per ct. 3.60	2.87	3.53	Per ct. 1.37	0.88	1.14	Per ct. 1.31	1.51	Per ct. 0.67	0.53
Oct. 14	1	All samples comparatively same; no signs of deterioration...	28.06	18.89	20.75	8.77	5.88	7.64	6.64	8.47	4.48	8.40
Oct. 15	2	Samples beginning to deteriorate; more marked in fresh and stored; preserved, fairly fresh.	19.47	23.17	3.01	9.46	7.07	12.72	1.67	8.19	1.15	1.35
Oct. 16	3	Slight odor noticeable; more marked in fresh and stored; samples were spoiling and becoming soft.	31.49	37.11	28.47	9.46	1.35	11.39	10.43	8.17	7.89	6.76
Oct. 19	6	Odor quite bad and samples spoiled; were soft and "mushy".	21.88	23.62	24.68	8.81	6.70	8.88	1.32	7.52	1.33	1.09
Oct. 20	7	Same as on 19th.	30.76	29.83	27.48	9.31	1.59	8.80	1.58	10.25	7.99	6.89
			6.71	5.39	5.80	9.64	10.31	10.76	12.13	10.48	8.89	7.68
			40.87	34.07	37.84	13.86	10.60	10.96	12.42	12.91	11.94	12.32
			6.34	6.28	7.22	2.09	2.01	2.31	1.19	1.32	1.86	3.13
			40.49	40.10	45.60	12.94	12.84	14.69	11.89	8.88	13.89	19.77

REPORT ON THE ADULTERATION OF DAIRY PRODUCTS.

By HERMANN C. LYTCHGOE, *Associate Referee.*

The referee, with the help of Messrs. Nurenberg and Marsh, assistant analysts of the Massachusetts State board of health, has made a study of the different methods for the preparation of milk serum and for the detection of calcium succate in cream. As a result of this work it is apparent that the provisional method for the preparation of milk serum needs no modification, but the method of Baier and Neumann for the detection of sucrose in milk or cream should be made provisional. The work done is embodied in the two following articles.

A COMPARISON OF METHODS FOR THE PREPARATION OF MILK SERUM.

The samples of milk used in this investigation were all milked in the presence of an inspector or an analyst of the Massachusetts State board of health and represented nearly all breeds of dairy cattle, particularly the Holstein, Ayrshire, Dutch Belted, and grade Holstein cows. The methods employed were the provisional (acetic acid) method, natural souring,^a calcium chlorid method,^b and asaprol method.^c The details of the methods other than the provisional methods are as follows:

Natural souring method.—Allow the samples to sour spontaneously and filter off the serum.

Calcium chlorid method.—Place 90 cc of milk in a flask, add 0.75 cc of calcium chlorid solution—sp. gr. 1.1375 (when diluted 1:10 this solution reads 26 on the immersion refractometer at 17.6° C.), shake thoroughly, close the flask with a cork carrying a glass tube to act as a reflux condenser, place in a boiling water bath for twenty minutes, cool to 20°, mix the condensed water and serum without shaking, and filter.

Asaprol method.—The precipitating solution is made by dissolving 30 grams of asaprol and 55.8 grams of crystallized citric acid in 1 liter of water. If the refraction of this solution is not 36.3 on the scale of the immersion refractometer at 20°, add water or citric acid to make it so. Mix equal volumes of the above solution and the milk, shake well, and filter.

In the accompanying table are the results of the refraction of the milk serum prepared from milk samples of known purity when two or more methods were applied to the same sample of milk. The asaprol method is by far the easiest of manipulation. It gives the clearest serum in the least time and shows the lowest refraction with the least variation. Unfortunately pure asaprol is very difficult to obtain, and, owing to the fact that it decomposes readily, it is not an easy matter to prepare different solutions that will give identical sera with the same sample of milk. The calcium chlorid method is the most difficult of manipulation and is liable to give a cloudy serum rather troublesome to read, but the results are lower than those obtained by the acetic acid method and not so variable. The natural souring method is too slow for ordinary use, but is valuable in the hot weather if the milk is nearly sour when it reaches the analyst. Four years' experience with the provisional method has shown it to be reliable, easy of manipulation, and to give concordant results.

^a Matthes and Müller, *Zts. öffentl. Chem.*, 1903, 10: 173.

^b Ackerman, *Zts. Untersuch. Nahr. Genussm.*, 1907, 13: 186.

^c Baier and Neumann, *Zts. Untersuch. Nahr. Genussm.*, 1907, 13: 369.

Refraction of milk sera from known purity milk of individual cows.

Method.				Method.			
Acetic acid.	Natural souring.	Calcium chlorid.	Asaprol.	Acetic acid.	Natural souring.	Calcium chlorid.	Asaprol.
46.2	47.7			43.0	43.2		
45.9	44.6		38.4	43.0	42.8		
45.8	44.0			43.0	42.3		
45.7	43.4	40.1	37.0	43.0	42.0		
45.6	43.5			43.0	41.7		
45.5	41.5	38.7	37.1	43.0	41.5		
45.1	41.5	39.0	36.8	43.0	41.4	39.1	36.4
44.9	41.5	39.8	36.1	43.0	41.1	38.4	36.6
44.8	43.8			43.0	40.9		
44.8	43.7	39.2	36.0	42.9	43.6		36.5
44.7	43.5		36.6	42.9	43.0		
44.6		38.5	36.6	42.9	41.4	39.0	36.7
44.5	45.0			42.8			37.4
44.8	42.8		35.7	42.7		38.8	
44.4	43.8		36.7	42.7		38.0	
44.3	43.0			42.6	41.3	38.1	36.1
44.3	42.8			42.5		39.3	36.3
44.3	42.3	39.0	37.0	42.5	41.5		
44.2	41.2	38.6	37.0	42.5		38.2	36.6
44.2	41.0			42.4	43.3		36.4
44.1	43.0	39.1	36.7	42.3		39.0	37.0
44.1	40.7	38.2	36.8	42.3	41.9		36.8
44.0	42.2	38.9		42.3	41.6	39.8	36.6
43.9	44.5			42.3	40.8	38.8	36.7
43.9	42.6			42.2	42.0		37.0
43.8	44.0			42.2	41.0		
43.8	44.0			42.1	44.0		36.8
43.8	43.0		36.7	42.1	43.7		36.6
43.8	41.6		37.5	42.0	41.0		
43.7	44.2		37.5	42.0	40.3	37.1	36.2
43.7	42.6			42.0	40.2	36.8	35.8
43.7	42.4			41.8	40.5		
43.7	41.5		36.9	41.7	40.9	38.2	36.1
43.6	43.0			41.7		38.0	
43.6	43.0	40.0	36.3	41.7	40.0	36.8	36.2
43.6	42.0	38.6	36.3	41.6	43.9		36.0
43.5	43.5			41.5	40.4	36.4	35.7
43.5	42.8			41.4	40.3	38.4	35.6
43.5	41.0	38.4	36.3	41.3	40.3		
43.4	43.1			41.2	40.0		
43.2	43.3			40.6	40.7	36.6	35.8
43.2	42.2			40.5	39.3		
43.2	41.8	38.9	36.6	40.4	38.3		
43.2	40.9		37.4	40.0	40.1		
43.0	43.7						

Mixed milk of known purity.

43.6	42.9	39.0	37.5	42.5	41.0	39.4	36.3
43.5	42.0	38.7		42.1	39.3		
43.4	40.8	38.2	36.7				

THE DETECTION OF CALCIUM SUCRATE IN MILK OR CREAM.

The calcium sucrate used in this investigation was prepared by adding 2.5 parts, by weight, of sugar to 1 part of quick lime slaked in 8 parts of water, allowing to settle, and decanting the supernatant liquid. The sample polarized at 17.3° V. in the 200 mm tube and its alkalinity was 1.86 normal.

Leffmann's method^a for the detection of calcium sucrate in cream, using sesame oil and hydrochloric acid as the reagents, was found to be satisfactory only in the presence of larger quantities than are necessary to thicken cream, therefore it was abandoned.

The method of Baier and Neumann^b was found to be entirely satisfactory for the detection of sugar, and is as follows:

^a Chem. Ztg., 1906, 30: 638.

^b Zts. Nahr. Genussm., 1908, 16: 51.

To 25 cc of milk or cream add 10 cc of a 5 per cent solution of uranium acetate, shake, allow to stand for five minutes and filter. If the filtrate is not clear pour it through the filter again. To 10 cc of the clear filtrate (in the case of cream use the total filtrate if less than 10 cc) add 2 cc of a cold saturated solution of ammonium molybdate and 8 cc of dilute hydrochloric acid (1 part of 25 per cent hydrochloric acid and 7 parts of water) shake well and place in a water bath at 80° C. for five minutes. If the sample is pure the solution will resemble a nickel sulphate solution, but if sugar is present it will be of a Prussian blue color. These different colors can be readily distinguished but it is advisable to compare with a standard blue solution made by adding a few drops of potassium ferrocyanid and 5 drops of 10 per cent hydrochloric acid to a solution of 1 cc of 0.1 per cent ferric chlorid in 20 cc of water.

Alkalinity of ash.—Evaporate 25 cc of cream to dryness, and burn to an ash in a muffle. Dissolve the ash in an excess of tenth-normal sulphuric acid, boil to expel the carbon dioxid and titrate back with tenth-normal sodium hydroxid, using phenolphthalein as the indicator. Express results as cubic centimeters of tenth-normal acid required to neutralize the ash of 100 grams of cream.

Determination of calcium.—Add acetic acid to the final solution from the above determination, heat to boiling, add 1 gram of sodium acetate and an excess of ammonium oxalate. Filter and wash the precipitated calcium oxalate with water, dissolve in hot dilute sulphuric acid, and titrate hot with tenth-normal potassium permanganate. The number of cubic centimeters of tenth-normal potassium permanganate multiplied by 0.0112 (4×0.0028) gives the percentage of calcium oxid in the sample.

The table appended shows the composition and reactions of pure and adulterated cream, using the Baier and Neumann method for calcium succrate. It is recommended that this method be distributed for criticism:

Results on pure and adulterated creams using the Baier and Neumann method for calcium succrate.

Pure cream.						Cream containing calcium succrate.					
Total solids.	Fat.	Ash.	Alkalinity of ash.	Calcium oxid.	Sucrose.	Calcium succrate added per liter.	Fat.	Ash.	Alkalinity of ash.	Calcium oxid.	Sucrose.
Per ct.	Per ct.	Per ct.	cc.	Per ct.	None.	cc.	Per ct.	Per ct.	cc.	Per ct.	Present.
51.20	45.2	0.36	7.6	0.073	None.	5	37.9	0.50	19.2	0.147	Present.
49.98	43.2	.35	6.0	.073	None.	2	37.9	.46	13.2	.130	Do.
47.86	42.8	.39	6.4	.062	None.	5	41.4	.39	11.6	.095	Do.
47.12	41.4	.37	6.8	.069	None.	4	42.8	.43	10.0	.101	Do.
.....	40.4	.43	8.8	.099	None.						
.....	40.4	.38	7.0	.092	None.						
47.00	39.2	.40	8.0	.091	None.		36.4	.29	16.0	.123	Do.
.....	39.2	.36	7.2	.085	None.		39.8		16.0	.143	Do.
45.02	39.6	.41	6.8	.083	None.		28.8		14.0	.135	Do.
43.08	36.8	.33	7.6	.094	None.		39.8		10.8	.130	Do.
42.80	37.2	.41	7.2	.086	None.		35.6		12.0	.141	Do.
42.75	36.8	.46	7.6	.083	None.						
MARKET SAMPLES.											

REPORT ON CEREAL PRODUCTS.

By E. F. LADD, *Associate Referee.*

During the past year considerable work was undertaken in our own laboratory upon cereal products, very little of which has as yet been completed. Therefore only a report of progress can be made. As the result of examinations made by A. S. Mitchell, chief of the St. Paul Food and Drug Inspection Laboratory, the following methods are suggested:

METHODS FOR ANALYSIS OF CEREAL PRODUCTS.

MOISTURE.

Dry a convenient quantity of the flour (approximately 5 grams) at the temperature of boiling water in a current of dry hydrogen or in vacuo until it ceases to lose weight.

ASH.

Char a convenient weight of the original sample (from 2 to 5 grams) in a platinum dish, in a muffle, at the lowest possible temperature until free from carbon. If carbon free ash can not be obtained owing to its fusibility, exhaust charred mass with water and proceed as under ash, Bulletin 107, page 38.

CRUDE FAT (ETHER EXTRACT).

Extract a convenient quantity of the product (from 4 to 5 grams) as dried in the determination of moisture with anhydrous, alcohol-free ether, for 24 hours (with fine flour the addition of an equal weight of clean dry sand is frequently necessary). Dry the extract at the temperature of boiling water until it ceases to lose weight.

NOTE.—Iodin numbers should only be obtained upon the ether extract after purification by solution in petroleic ether, but are best made upon the petroleum ether extract.

SOLUBLE CARBOHYDRATES (AS DEXTROSE).

Weigh 16 grams of flour into a 500 cc flask. Add 200 cc of water. Shake occasionally during one-half hour. Filter through a dry folded filter. To 50 cc of the filtrate add 5 cc of concentrated hydrochloric acid. Place the flask in water and invert at 70° C. for ten minutes. Cool, neutralize, and bring to 100 cc. Filter. Determine the reducing sugars with Fehling solution, by the official method, as described in Bulletin 107, calculating the reducing sugars as dextrose.

CRUDE FIBER.

Determine the crude fiber in 2 grams of flour by the official method (Bul. 107), filtering through linen in a Büchner funnel.

DETERMINATION OF MOIST GLUTEN.

Dough up 30 grams of flour with 18 cc of water conveniently in an 8-ounce mortar. Weigh off 16 grams of dough equivalent to 10 grams of flour. Place in water at room temperature for one hour and carefully wash out the starch over bolting cloth or a fine horsehair sieve. After expressing all globules of water, weigh the moist gluten upon a watch glass. Dry in a desiccator for 24 hours and complete drying in water oven.

ACIDITY IN FLOUR.

Weigh 18 grams of flour into a 500 cc Erlenmeyer flask and add 200 cc of distilled water, previously freed from carbon dioxid by boiling in tin. Place the loosely stoppered flask in a water bath kept at 40° C. for 10 minutes, shaking repeatedly. Remove the flask and allow it to stand, with occasional shaking, at room temperature for one hour. Filter upon a dry folded filter, rejecting the first 10 cc and receiving the succeeding 100 cc in a graduated flask. Titrate the filtrate with twentieth-normal sodium hydroxid, using carefully neutralized phenolphthalein in alcohol as an indicator. Each cubic centimeter of twentieth-normal sodium hydroxid represents 0.05 per cent of acidity as lactic acid.

NOTE.—Results obtained with flour at temperatures of 15°, 20°, and 25°, respectively, indicate that the acidity in the solution increases with the temperature. The method outlined seems to give the maximum acidity.

TOTAL NITROGEN IN FLOUR.

Determine the total nitrogen in 2 grams of flour according to the official method, preferably the Gunning method, Bulletin 107, page 7. The nitrogen times 6.25 gives total proteins.

GLOBULIN AND ALBUMEN (EDESTIN AND LEUCOSIN) AND AMID NITROGEN.

Weigh 5 grams of flour into a 500 cc Erlenmeyer flask. Add 250 cc of sodium chlorid solution 1 per cent. Stopper and shake thoroughly. Let stand, with occasional shaking, for three hours. Filter on dry paper. Evaporate 100 cc of the filtrate to small volume in a Kjeldahl digestion flask with 5 cc of sulphuric acid. Add remainder of the

sulphuric acid and determine the nitrogen by the Gunning method. To a second 100 cc of the filtrate add 5 cc of phosphotungstic acid, 20 per cent solution; shake thoroughly, allow to settle, and filter by decantation. Wash slightly with water. Concentrate the filtrate with 5 cc of sulphuric acid in Kjeldahl flask and determine the nitrogen as amid.

Deduct the amid nitrogen from the nitrogen found in the first fraction to obtain the nitrogen as globulin and albumen. This figure times 6.25 gives globulin and albumen.

ALCOHOL SOLUBLE PROTEINS (GLIADIN).

Weigh 4 grams of flour into a 500 cc Erlenmeyer flask, add 200 cc of alcohol 0.90 sp. gr. Shake occasionally during three hours. Let stand 12 hours. Filter through a dried filter. Evaporate the alcohol from 100 cc of the filtrate after the addition of 5 cc of sulphuric acid and determine the nitrogen as alcohol soluble nitrogen. This figure, less the amid nitrogen, gives the alcohol soluble proteid nitrogen or gliadin.

GLUTENIN (DETERMINATION BY DIFFERENCE).

Deduct from the total nitrogen the salt soluble nitrogen plus the gliadin. This times 6.25 gives the glutenin.

GLIADIN BY POLARIZATION (METHOD OF SNYDER).

Weigh 15.97 grams of flour into a 300 cc flask. Add 100 cc of 0.90 sp. gr. alcohol. Shake at intervals during three hours and let stand overnight. Filter through a dry folded filter. Polarize in a 220 mm tube. Precipitate the proteids in 50 cc of the filtrate with 5 cc of Millon's reagent. Shake, filter, and polarize the filtrate in a 220 mm tube. Add 50 per cent to the reading and deduct the sum from the first reading. This difference times 0.2 gives the per cent of nitrogen as gliadin.

FAT DETERMINATION (BASSETT).

An effort was made to discover a method whereby the time for determining fat and moistures in cereal samples, especially flour, could be much shortened and without a sacrifice of accuracy. H. P. Bassett, of the North Dakota laboratory, was assigned some work along this line, the results of which are embodied in the following:

Fat in flour has been determined usually by the method given by Leach, which is outlined so as to be applicable to all food and feeding stuffs. However, in making fat determinations on flour by Leach's method considerable time is required, and unless special precautions are taken the analyst could never check himself. This, in any method, indicates inaccuracy. In examining the difficulties which might arise to affect this method, it was especially noted that oxidation might take place in drying the flour in a hot-water oven, as is generally practiced, since the fat in the flour is in a fine state of division, which gives the most favorable conditions for oxidation. Again, the special precaution of removing the last trace of moisture from the flour seemed to be an unnecessary point when the ether, as generally employed, contained probably ten times more water than was found in the dried flour.

The extraction by the Leach extractor is also slow, requiring sixteen hours, and in apparatus arranged in such a manner that it can not always be run with safety overnight. This means, then, three full working days before a determination can be made—one for the moisture determination and two for the fat determination.

In order to avoid these difficulties, the following method was developed:

Ten grams of flour were weighed into a tared gooch crucible, then placed in the ordinary gooch funnel, which was inserted into a rubber stopper in the top of a low bell-jar, which rested upon a ground-glass plate. Under the bell-jar and directly under the gooch funnel was placed a second glass plate to avoid the possibility of getting vaseline on the bulb, which was to catch the filtrate, vaseline being used to make an air-tight joint between the bell-jar and ground-glass plate. The gooch was now filled with ether six times, each time drawing off with the filter pump. The ether extract was collected in a bulb similar to those with a Soxhlet apparatus. This bulb was then removed and connected with a Liebig's condenser, and the ether distilled off with a 32 candle-power incandescent bulb, this being used as it avoided the possibility of the vapors of ether catching fire, and also has the additional advantage of not being so hot

as to easily burn the fat. The residue in the gooch crucible is now dried in an air oven and weighed, the loss in weight being equal to the fat and moisture. The fat having been determined, the moisture is easily obtained by difference.

The results by this method, however, are considerably higher than by the Leach method—often twice as much—but there is no difficulty in the analyst duplicating his results. Following are some of the figures obtained by this method:

Comparison of methods for fat determinations.

Number of experiment.	New method.		Leach method.	
	1.	2.	1.	2.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
1.	1.26	1.24	0.47	0.45
2.	1.70	1.71	1.21	1.23
3.	2.29	2.30	1.83	1.87
4.	1.16	1.22	.68	.70
6.	1.08	1.12	.65	.53
7.	2.66	2.66	1.17	1.27
8.	1.07	1.07	.60	.62
9.	1.32	1.36	1.30	1.28
10.	2.14	2.18	2.37	2.28
11.	.97	.99	.70	.65
12.	1.05	1.02	.65	.65
13.	3.05	3.00	2.43	2.52

DISCUSSION OF RESULTS.

Comparing the results by the two methods, it is noticed at once that none of those by the new method checks those made by the old method, and it was thought at first that there might possibly be an error on account of the moisture present in the flour while extracting, the tests by the old method being carried out on dry flour. A large amount of moisture would probably cause some of the sugar-like substances to be extracted. In order to test this point, the following experiments were performed: Ten grams of flour were weighed out in a gooch crucible as before and placed in a large-mouthed bottle, which was closed with a two-hole rubber stopper. Carbon dioxid which had been dried over concentrated sulphuric acid was conducted through the bottle, the same being arranged in a water bath and heated for four days under these conditions. This was then extracted with ether, according to the new method. The results obtained checked exactly those extracted without drying.

Results obtained by new method on dried and fresh flours.

No.	Fat in dried flour.	Fat in fresh flour.
	<i>Per cent.</i>	<i>Per cent.</i>
1	1.21	1.24
2	1.22	1.26
3	1.24	1.23
4	1.24	1.22

The next point considered was to determine if flour dried in carbon dioxid, under these conditions, would give the same per cent of fat as by the new method or would check the old method. Therefore, 2 grams of flour were weighed out in a small test tube with a hole in the bottom, which was closed by an asbestos plug. This was then placed in the bottle described and dried for four days, after which it was extracted by the Leach method, the following results being obtained:

Fat determinations, drying with carbon dioxid.

New method.	CO ₂ -dried flour by old method.
1.26	1.21
1.24	1.22
1.23	1.21
1.22	1.19

It is evident that when dried in carbon dioxid the flour does not undergo any oxidation, while from these results the point seems almost, if not completely, proven that it does undergo oxidation when dried in the open air, and for this reason it is hard to obtain results that check.

Numerous determinations have been made by this method and it has given perfect satisfaction as well as being extremely rapid. Time may be saved, as the gooch crucible does not need a new pad every time, the same one being used for at least fifteen determinations by simply knocking out the extracted flour when through. Further, the flask containing the fat may be used six or eight times without cleaning, where a number of determinations are being made.

This work was carried out with ether as the solvent, but chloroform, acetone, or benzin may also be used, similar results being obtained. The following results were obtained:

Fat determinations by the new method, using different solvents.

No.	Ether.	Chloroform.	Acetone.	Benzin.
1	1.24	1.26	1.29	1.20
2	1.26	1.27	1.26	1.21
3	1.23	1.25	1.27	1.23
4	1.22	1.29	1.28	1.22

These results become of value on account of the cost, chloroform and benzin being much cheaper than ether or acetone.

In some cases feed and foodstuffs are not well ground, nor capable of being ground as fine as flour. These of course would not extract by the above method readily, but may be extracted by means of the Soxhlet apparatus instead of the gooch crucible. The Soxhlet apparatus was used on flours and the results check those made with the gooch crucible very closely.

Either one of the solvents named may be used instead of ether, as a larger amount of solvent is required under such conditions, and unless special precautions are taken considerable loss may take place.

Comparison of results on fat, using a gooch and the Soxhlet apparatus.

Gooch crucible.	Soxhlet apparatus.
1.24	1.24
1.26	1.26
1.23	1.27
1.22	1.28

MOISTURE DETERMINATION.

The moisture, as stated in the preceding method, may also be determined by drying the residue in a hot-water oven and then weighing the crucible and residue, the loss being equal to the weight of the fat and moisture from which the moisture may be determined. The following results were obtained and will be compared with the old method by Leach:

Comparison of moisture determinations by two methods.

New method.	Old method.	
<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
8.60	8.76	8.66
8.80	8.76	8.66
8.88	8.76	8.66
8.92	8.76	8.66

The results here, however, are not so close as in the fat determinations, but out of the numerous determinations that have been carried out in this laboratory the results have checked to within 0.2 to 0.3 per cent, and, in the majority of cases, within a hundredth of a per cent. The following table shows a few results which will give an idea of the accuracy of the method:

Duplicate moisture determinations by new method showing degree of accuracy.

Sample.	Per cent.	Per cent.
1	12.64	12.76
2	12.36	12.56
3	11.08	11.06
4	11.40	11.84
5	12.80	12.06
6	11.40	11.30
7	11.72	11.80

This method has been a means of saving much time, since a large number of such determinations were made during the flour investigation of the past year.

Considerable work was also undertaken in the study of the gluten and protein content of the flour, and a large number of methods were tested. Some results of special interest were secured, but owing to the illness of the assistant having this work in charge only progress in this direction can be reported.

RECOMMENDATION.

It is recommended that for the coming year special attention be given to testing methods for the separation of the gluten constituents of flour, tests being made upon the several grades, as patent, first and second clears, and upon flours produced from different varieties and types of wheat.

REPORT ON VEGETABLES (CANNED PEAS).

By W. L. DUBOIS, *Associate Referee.*

The work on this subject during the last year has been confined to the examination of canned peas for the purpose of distinguishing soaked peas from those canned when fresh. Such a distinction of course is made with quite a degree of certainty by a

simple examination of the physical appearance of the goods, noting especially the maturity and firmness of the peas and the consistency of the liquor. Soaked peas usually appear more or less broken and mashed and the most matured show well developed cotyledons and are packed in a liquor which is cloudy and starchy in appearance.

The maturity of the peas, however, can not be taken as conclusive evidence that the same have been soaked, because many well developed peas, very similar in appearance to those soaked before canning, are packed as numbers 4 and 5, Early June and Telephone peas, and are not soaked. Neither can the appearance of the liquor be finally relied upon, since the most mature, fresh peas are sometimes found in a liquor which is not clear and is more or less starchy; hence it is desirable to obtain data which would substantiate conclusions drawn from the physical appearance of the goods. To this end 73 miscellaneous samples of peas have been examined by the referee and on all these the weight of the liquor and drained substance, and the percentage of water in the drained substance were determined. These determinations alone are sufficient to distinguish the fresh peas and some of the more succulent grades from the soaked goods, the chief difficulty arising in differentiating between the soaked goods and the more matured peas put up in the fresh state. In the water content the latter did not differ very widely from the soaked peas. As will be seen from the table, the average content of water in 24 samples of soaked peas is 71.98 per cent, and the average of 18 samples of Early June and similar grades is 77.52 per cent. The highest moisture content of the soaked peas, however, exceeds that of the driest of the Early June peas, so that there is an overlapping of the results which makes it impossible to pronounce a conclusive opinion from these determinations alone.

More definite conclusions, however, may be drawn by also determining the crude starch. For this determination 15 grams of the ground drained material were hydrolyzed by hydrochloric acid according to the official method and all copper-reducing substance calculated as starch. The average of 16 results on soaked peas gave 14.45 per cent, the highest figure being 18.19 per cent and the lowest value 11.08 per cent, while the average starch content of 11 samples of matured peas canned in the fresh state was 10.87 per cent. Here again is an overlapping, the lowest results on the soaked peas, 11.08 per cent, being below the highest value obtained on the fresh grade, 14.38 per cent. This last sample, however, was probably misbranded, as will appear later. The average starch content on soaked peas, as far as determined, is approximately 4 per cent higher than that of Early Junes and those of similar quality. There is some difference, furthermore, in the specific gravity of the two grades, that of Early Junes running from 1.10 to 1.14, whereas the values obtained for soaked peas vary from 1.12 to 1.16. Taking all these figures into consideration, it seems possible by the determination of the water, starch, and specific gravity of the drained substance to obtain values which will supplement the conclusions drawn from the physical appearance of the goods.

The table gives in detail the results obtained. Samples numbered 81 and 82 are interesting in furnishing a test of the method suggested. These samples were labeled Early June peas, but both had the appearance of having been soaked. These were run at the same time as numbers 75 to 80, inclusive, and it will be seen how the starch content compares with the other samples labeled in the same way. By these values alone and the appearance of the goods it would be quite safe to conclude that they had been soaked. There is also a difference in the specific gravity, which is somewhat higher than in the other samples. This conclusion is further strengthened by the amount of water which is less than in the other samples.

The work seems to justify further investigation along the same line by the succeeding referee.

Examination of canned peas to distinguish soaked goods.

FRESH PEAS.

Number.	Grade and appearance.	Liquor.	Drained substance.	Specific gravity.	Crude starch.	Protein.	Water.
		Grams.	Grams.		Per c.	Per c.	Per c.
1	Large, firm, not succulent.....	194	398				78.61
2	First quality, sifted.....	201	389				82.68
3	Peas quite soft.....	193	400				81.87
4	Fairly soft.....	208	387				82.61
5	Soft, sifted, Telephone peas.....	196	396				82.94
6	Mealy, fancy, sifted, large peas.....	223	360				78.35
7	Fancy, sifted, sweet, good.....	187	378				86.84
8	do.....	197	390				78.53
9	do.....	222	365				78.87
10	Extra fancy, sweet, good.....	205	360				87.04
11	Extra fancy, good.....	190	380				86.87
12	Extra fancy, sweet, good.....	190	380				86.95
	Surprise peas:						
13	No. 1 small.....	190	395				87.20
14	No. 2.....	220	383				86.57
15	No. 3.....	238	362				86.57
16	No. 4.....	202	400				82.52
17	No. 5.....	216	382				81.91
	Alaska peas:						
18	No. 1.....	255	332				86.72
19	No. 2.....	245	344				82.37
20	No. 3.....	245	357				78.23
21	No. 4.....	220	387				76.34
22	No. 5.....	240	357				74.28
	Admiral peas:						
23	No. 1.....	215	375				86.80
24	No. 2.....	203	387				84.59
25	No. 3.....	227	369				84.30
26	No. 4.....	220	369				80.83
27	No. 5.....	219	373				78.50
28	First quality; a few old.....	224	364				83.63
29	Good quality; a few old.....	209	381				79.29
30	Early June.....	205	380				79.59
31	Large, firm, about same as No. 30.....	197	356				86.73
32	Tom Thumb, very small, excellent.....	210	359				85.99
33	Petit Pois, small.....	238	334				85.99
34	Sifted Little Gem, small.....	233	353				85.91
65	Early June, good quality.....	215	383	1.12-1.16	13.88		70.79
66	Early June Extra No. 7, fair.....	265	305	1.10-1.14	12.13		74.55
67	Telephone, some broken and soft.....	218	374	1.10-1.14	12.15		74.62
68	Early June, a little mushy.....	232	372	1.10-1.16			75.10
69	Early June, appear soaked.....			1.10-1.14			
70	Early June, cloudy and starchy, firm.....			1.10-1.14			
71	Early June, cloudy and starchy, old.....			1.10-1.16			
72	Early June, poor quality, appear soaked.....			1.12-1.16			
73	Early June, good quality.....			1.08-1.16			
74	Early June, cloudy, poor, many broken.....			1.10-1.16			
75	Early June, firm, large, many hard.....	232	351	1.08-1.14	10.55		Lost.
76	Early June, medium size, good quality.....	229	366	Below 1.10	7.37		82.30
77	Early June, medium size, fair quality.....	250	335	1.08-1.16	12.00		75.45
78	do.....	Lost.	Lost.	Below 1.10	6.63		84.04
79	Early June, large, mealy, many hard.....	200	378	1.08-1.12	9.34		78.88
80	do.....	189	386	1.08-1.14	7.20		77.89
81	Early June, large, mealy, appear soaked.....	299	277	1.08-1.14	14.38		74.84
82	Early June, appear soaked.....	252	294	1.10-1.14	13.95		73.21

Examination of canned peas to distinguish soaked goods—Continued.

SOAKED PEAS.

Number.	Grade and appearance.	Liquor.	Drained substance.	Specific gravity.	Crude starch.	Protein.	Water.
		<i>Grams.</i>	<i>Grams.</i>		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
35	Soaked, some mushy.....	235	359	1.12-1.18			70.71
36	do.....	255	335	1.12-1.16	14.05		73.43
37	do.....	243	324	1.12-1.16			71.67
38	do.....	294	274				74.36
39	do.....	231	344	1.10-1.16	13.70		62.07
40	do.....	215	353	1.10-1.16			74.92
41	do.....	187	405	1.12-1.16			73.68
42	do.....	211	380				70.91
51	Liquor very cloudy, soaked.....	274	306		11.06	6.87	71.84
52	Soaked, very poor, mushy.....	222	280		18.19	6.69	73.84
53	Soaked, liquor very cloudy.....	177	401		14.23	6.56	72.46
54	Soaked, sample poor.....	152	418		12.83	6.13	74.66
55	Soaked, cloudy, peas fair.....	213	368		13.54	6.69	69.67
56	Soaked, cloudy, broken, and soft.....	175	408		14.47	6.44	71.97
57	Soaked, mushy.....	187	390	1.12-1.16	13.96	6.06	74.10
58	Soaked, many broken.....	267	300	1.12-1.16	14.79	7.31	71.15
59	Soaked, very mushy.....	245	330	1.12-1.16	14.31	6.63	72.67
60	Soaked, fairly firm.....	253	341	1.12-1.16	14.77	6.19	73.05
61	Soaked, some soft, color fair.....	197	385	1.12-1.16	15.25		71.00
62	Soaked, many poor.....	250	303	1.12-1.16	16.04		71.70
63	Soaked, good, some black.....	214	323	1.12-1.16	15.77		71.06
64	Soaked, somewhat mushy.....	304	258	1.12-1.16	14.34		73.62

REPORT ON THE SEPARATION OF MEAT PROTEIDS.

By P. F. TROWBRIDGE, *Associate Referee.*

During the past year no cooperative work has been asked for on the separation of meat proteids, but considerable work has been done at the Missouri experiment station, in connection with the general meat investigations, in the examination of cold water extracts of meat from composite samples of various wholesale cuts of steers of different ages and of different degrees of fatness.

In the preparation of cold water extracts the general plan as proposed by Grindley and Emmett ^a was followed with slight modifications.

With lean samples exactly 100 grams and with fat samples 150 grams were taken; in each case being distributed through eighteen beakers in about equal portions. The sample in each beaker was thoroughly mixed with 3 to 5 cc of cold neutral nitrogen-free distilled water and then with 50 cc of the same water. The mixture was allowed to stand about thirty minutes, with frequent agitation, and then poured through previously wetted filters. Each time the residue which was poured on the filter was returned to the beaker. To the residue in the beaker 25 cc of water were added and the meat residue thoroughly stirred and again filtered. This washing was continued till 225 cc of water were used; then the whole residue was transferred to the filter and washed twice with 10 to 15 cc of water each time.

The filtrates were combined, the flasks rinsed, and the total volume made up to 5,000 cc. This cold water extract was carefully mixed without undue agitation and filtered through a dry filter just before the aliquot portions were taken for analysis. (In all filtrations the funnel was made to touch the sides of the flask or beaker, so as to limit the amount of spontaneous coagulation.)

Triplicate samples were taken for analysis as follows:

a, b, c.—100 cc for total soluble nitrogen representing 2 grams of lean and 3 grams of fat sample.

d, e, f.—100 cc for total solids and ash representing 2 grams of lean and 3 grams of fat sample. * * *

m, n, o.—200 cc for coagulable nitrogen representing 4 grams of lean and 6 grams of fat sample.

^a J. Amer. Chem. Soc., 1905, 27: 661.

p, q, r.—200 cc for total albumose nitrogen; filtrate from *m, n, o*, representing 4 grams lean and 6 grams of fat sample.

s, t, u.—200 cc for total amido acid nitrogen representing 4 grams of lean and 6 grams of fat sample.

Samples *a, b, c* were transferred to 500 cc nitrogen flasks for the direct determination of nitrogen by the Kjeldahl-Gunning method.

Samples *d, e, f* were evaporated to dryness on the water bath, then dried to constant weight in air bath at 103°, and finally ashed at a dull red heat.

Samples *m, n, o* were treated with a slight excess of moist magnesium carbonate,^a evaporated to about 30 cc, filtered and washed with hot water to which a little moist magnesium carbonate had been added. The precipitate and filter were transferred to 500 cc nitrogen flasks and the coagulum adhering to the sides of the beakers was removed with hot sulphuric acid and transferred to the corresponding flask. The nitrogen was determined in the usual manner.

The filtrates from *m, n, o, (p, q, r)* were concentrated to about 10 cc in small beakers and acidified with 1 cc of 50 per cent sulphuric acid, diluted to 30 cc and to each 50 grams of pure crystallized zinc sulphate were added. The mixture was then heated upon the water bath until the complete solution of the zinc sulphate took place. If too much zinc sulphate crystallized out upon cooling a little water was added, care being taken to have only a slight excess above saturation. The contents of the beakers were filtered through filters previously wet with a saturated solution of zinc sulphate slightly acidified with sulphuric acid. After the filtrate had completely drained through, the beaker and filter were washed three times with the saturated zinc sulphate solution, allowing the washing to drain completely before adding the next washing. The filter and precipitate were transferred to nitrogen flasks and each beaker washed with water and sulphuric acid, the washings being rinsed into the corresponding flask. If care has been used in avoiding an excess of zinc sulphate crystals there will be no trouble with bumping during the digestion for the determination of the albumose nitrogen.

Samples *s, t, u* were treated as for coagulable nitrogen. The filtrates from the coagulum, not to exceed 30 cc, were rinsed into 100 cc graduated flasks, 15 grams of sodium chlorid were added and dissolved by warming gently. The flasks were placed in the ice box until cooled to 15° C. A 24 per cent solution of tannic acid was made up, filtered, and cooled in the ice box. When both solutions were cooled, 30 cc of the tannic acid solution were added to each 100 cc flask, which was then filled to the mark with cold water; the contents of the flask were thoroughly mixed and allowed to remain in the ice box over night. The following morning they were filtered rapidly and 50 cc of the filtrate transferred to nitrogen flasks for the determination of the amido acid nitrogen. With all of the determinations blanks were made to correct for the nitrogen in the reagents. From these data the nitrogen present as peptone nitrogen was calculated.

The main purpose in the examination of the water extracts of the fresh meats has been to see if age of animal or condition of fatness has any influence upon the amount of water-soluble material, or upon its composition, also to what extent there is a variation in different parts of the animal. To this end the samples have been handled as nearly as possible in the same manner and with the same treatment after slaughtering. So far eight animals have been slaughtered and analyzed, but the data are still insufficient to admit of any general conclusions, and the present paper is to be regarded only as a report of progress. A tabulation of a few of the results is appended, selecting those for the round and rump, the rib and loin cuts of the first four animals slaughtered.

^a Prepared by precipitating magnesium chlorid with sodium carbonate, heating, filtering, and washing until no chlorids remained in the filtrate.

Distribution of nitrogen in cold-water extracts of wholesale cuts, free from bone, from four different steers.

Sample.	Total solids.	Total nitrogen.	Coagu- lable nitrogen.	Albu- mose nitrogen.	Peptone nitrogen.	Amido- acid nitrogen.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Steer No. 18: ^a						
Round and rump	4.979	0.574	0.287	0.037	0.040	0.210
Rib	4.627	.540	.271	.039	.053	.177
Loin	4.778	.550	.263	.052	.064	.171
Steer No. 121: ^b						
Round and rump	4.583	.528	.253	.142	None.	.202
Rib	3.490	.413	.192	.010	.053	.158
Loin	3.631	.409	.195	.042	.010	.162
Steer No. 505: ^c						
Round and rump	5.116	.587	.273	.035	.007	.272
Rib	4.117	.470	.199	.029	.021	.221
Loin	4.593	.534	.235	.041	.029	.229
Steer No. 503: ^d						
Round and rump	5.389	.627	.294	.071	.036	.226
Rib	4.629	.530	.254	.033	.034	.209
Loin	4.348	.501	.224	.071	.015	.181

^a Grade Shorthorn steer, 3 years old and extremely thin.

^b Grade Shorthorn steer, 3 years old and moderately fat.

^c Grade Hereford steer, 1 year old and moderately fat.

^d Grade Hereford steer, 1 year old and in fair condition as a stocker, much better condition than No. 18.

^e The albumose precipitate was washed only once; results too high.

Distribution of nitrogen in cold-water extracts of the lean of wholesale cuts (bone and fat hand separated) from three steers.^a

Sample.	Total solids.	Total nitrogen.	Coagu- lable nitrogen.	Albu- mose nitrogen.	Peptone nitrogen.	Amido- acid nitrogen.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Steer No. 121:						
Round and rump	5.736	0.671	0.320	^b 0.173	None.	0.258
Rib	4.548	.550	.251	.013	0.073	.213
Loin	5.398	.647	.306	.040	.041	.260
Steer No. 505:						
Round and rump	6.153	.710	.332	.039	.009	.330
Rib	4.939	.565	.240	.032	.025	.268
Loin	6.169	.719	.315	.054	.038	.312
Steer No. 503:						
Round and rump	5.980	.699	.330	.074	.040	.255
Rib	4.930	.567	.276	.028	.037	.226
Loin	5.510	.643	.301	.083	.023	.236

^a Steer No. 18 excluded as it was the first one slaughtered, and the hand-separated fat was weighed separately but was ground with the lean for analysis.

^b Albumose precipitate washed but once.

Distribution of nitrogen in cold-water extracts of wholesale cuts, free from bone and fat,^a of four different steers.

Sample.	Total solids.	Total nitrogen.	Coagu- lable nitrogen.	Albu- mose nitrogen.	Peptone nitrogen.	Amido- acid nitrogen.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Steer No. 18:						
Round and rump	5.740	0.662	0.331	0.043	0.046	0.242
Rib	5.645	.659	.331	.048	.064	.216
Loin	6.103	.703	.336	.066	.083	.218
Steer No. 121:						
Round and rump	6.072	.700	.335	^b .188	None.	.268
Rib	5.612	.664	.309	.016	.085	.254
Loin	5.913	.685	.327	.070	.017	.271
Steer No. 505:						
Round and rump	6.631	.761	.354	.045	.009	.353
Rib	6.030	.688	.291	.042	.032	.323
Loin	6.705	.779	.343	.059	.043	.334
Steer No. 503:						
Round and rump	6.167	.717	.336	.061	.041	.259
Rib	5.453	.624	.299	.039	.040	.246
Loin	5.903	.680	.318	.096	.020	.246

^a The weight of the actual amount of fat (ether-soluble) is deducted from the weight of the cut. This reduces all cuts to a fat-free basis.

^b Albumose precipitate washed but once.

In the first table it will be noticed that if the data concerning the albumoses and peptones are omitted, the round and rump cuts in every case give higher figures than the other two cuts. When the hand-separated fat is eliminated we find less variation; the round and rump cut gives higher results than the rib but is equaled or slightly surpassed in a few cases by the loin. When the fat is entirely eliminated, as shown in the third table, the difference is still less. However, in only one case does the rib cut (steer 18, coagulable nitrogen) give as high results as the round and rump cut; and in another case (steer 503, amido-acid nitrogen) as high as the loin. In six cases the loin cut gives higher results than the round and rump cut. In general, steer No. 505 gives the highest results, especially in the case of the amido-acid nitrogen, there being only one exception, namely, the coagulable nitrogen in the rib cut on the fat-free basis. A further discussion of these results will not be attempted at this time.

For the purpose of making an extended study of the composition of beef extract there was prepared at the Missouri station (at the time of slaughtering) a cold-water extract ^a from a 5-kilo sample of the round of each animal. The filtered extract was coagulated upon the water bath, filtered, and concentrated with one or two filtrations as the concentration proceeded. The extracts have been finally concentrated to a semisolid mass, in which condition they appear to remain in a state of perfect preservation. The life history of the animals from which these extracts have been prepared is known and during the next year cooperation in the examination of these extracts will be requested, to determine the composition of pure beef extract and to learn to what extent variation may be expected.

The president announced the following membership for Committee B on recommendation of referees: Messrs. B. B. Ross, R. W. Thatcher, A. S. Mitchell, Paul Collins, and W. D. Bigelow.

The following committee was appointed to wait upon the Secretary of Agriculture and the Assistant Secretary and invite them to address the convention: Messrs. M. E. Jaffa, J. M. Bartlett, and W. A. Withers.

On motion by Doctor Wiley, the vote on the amendments to the constitution was made special order for 12 o'clock, or following the presidential address, on Friday.

REPORT ON PRESERVATIVES.

By W. D. BIGELOW, *Referee*.

SALICYLIC ACID.

RAPID DETERMINATION OF SALICYLIC ACID.

The methods of the association for the quantitative determination of salicylic acid are long and tedious because of repeated extraction with immiscible solvents. An attempt was made to simplify these methods by extracting a certain volume of the food, or an aqueous extract thereof, by means of a definite volume of solvent, evaporating to dryness an aliquot portion of the solvent used, and determining the salicylic acid in the residue. The total amount can then be calculated by a factor to be determined by experimental work.

It is, of course, necessary that the solvent, under certain conditions to be adopted, should extract a uniform amount of salicylic acid uncontaminated by substances that

^a Trowbridge and Grindley, J. Amer. Chem. Soc., 1906, 28: 472.

would interfere with the reaction by which the salicylic acid should finally be estimated in the residue. Owing to its rapidity and convenience, the ferric chlorid reaction has usually been employed for determining the amount of salicylic acid present. It is, therefore, important that the solvent employed should not extract tannin from the food.

In order to determine what solvents should be most advantageously employed as far as delicacy of reaction and freedom from tannin or other interfering bodies is concerned, Mr. Charles S. Ash extracted 50 cc portions of claret containing salicylic acid in amounts varying from 0.025 mg to 0.5 mg and treated the residue obtained by evaporation of the solvent with ferric chlorid in the usual manner. The results are given in the following table:

Comparative efficiency of solvents on salicylic acid dissolved in 50 cc of claret (Ash).

Milli-grams salicylic acid present.	Ether.	Chloroform.	Ether and petroleum ether.	Dichloroacetylene.	Trichloroacetylene.	Ether and hydrogen peroxid.	Ether, residue extracted with—		Petroleum ether.	Carbon bisulphid.	Carbon tetrachlorid.	Toluene.
							Carbon tetrachlorid.	Petroleum ether.				
0.500	None.	Good.	Faint trace.	Good..	Good..	Good..	Good.	Good....	Faint trace.	Good.	Good..	Good.
.250		Good.	None..	Fair...	Good..	Faint	Good.	Faint....	None..	Good.	Good..	Good.
.100		Good.		Trace	Trace	Trace	Fair..	Nothing.		Trace	Good..	Good.
.050		Good.		Trace?	None..	None..	None.			(?)	Good..	Good.
.025		Good.		None..							Trace ?	Good. Good trace.

Mr. Ash found that the first five solvents given in the table extract tannin in the order in which they are mentioned—that is, ether extracts the greatest amount and trichloroacetylene the least. The last three solvents—carbon bisulphid, carbon tetrachlorid, and toluene—do not extract tannin, and the ferric chlorid reaction in the residue obtained by them from wine is clear and characteristic of ferric salicylate. It will be noted that the residue from the ether extraction gave no reaction whatever with ferric chlorid. This was due to the presence of tannin, which entirely obscured the reaction. With chloroform much better results were obtained, but even here the reaction was partially obscured by tannin, which was also true of the residue from dichloroacetylene.

The data given in the column headed "Ether and hydrogen peroxid" were determined by oxidizing with ammonia and hydrogen peroxid the residue obtained by evaporating the ether extract. This destroys the tannin and also partially converts benzoates and saccharin when present into salicylic acid. The salicylic acid was then again extracted with ether, the ether extract evaporated, and the residue tested with ferric chlorid. As previously stated, the greatest freedom from interfering substances attended the use of carbon tetrachlorid and toluene, the latter appearing to extract slightly more salicylic acid than the former, and thus affording a better test in the presence of a small amount of that substance. Chloroform was also very satisfactory, being inferior to carbon tetrachlorid and toluene in respect of dissolving interfering substances, though apparently slightly superior in the amount of salicylic acid extracted.

Extraction of salicylic acid from different wines.

[Salicylic acid found.]

BY MEANS OF CARBON TETRACHLORID.

Salicylic acid added.	Angelica.			Sherry.			Port.			Claret.			Dry white wine.		
	Colorimetrically.	By weight.	By titration.	Colorimetrically.	By weight.	By titration.	Colorimetrically.	By weight.	By titration.	Colorimetrically.	By weight.	By titration.	Colorimetrically.	By weight.	By titration.
Mgs.	Mgs.	Mgs.	Mgs.	Mgs.	Mgs.	Mgs.	Mgs.	Mgs.	Mgs.	Mgs.	Mgs.	Mgs.	Mgs.	Mgs.	Mgs.
100	63.2	63.4	60.8	60.6	61.6	60.2	60.0	60.2	60.0	49.4	49.6	50.4	51.0	49.6	49.2
50	30.6	30.6	30.6	30.6	31.6	32.4	29.8	30.4	30.8	24.4	24.8	24.2	24.6	25.4	24.6
25	14.6	14.6	15.2	14.8	15.2	15.8	14.8	14.6	14.8	13.4	11.8	12.0	13.6	12.4	12.4
10	6.0		6.2		6.2		6.0		6.0			5.6	5.2		5.6
5	3.0			3.2			3.0			2.6			2.4		
2.5	1.6			1.6			1.6								
0		4.4	1.4		4.8	1.4		5.0	1.4		3.8	0.8		3.8	1.2

BY MEANS OF TOLUENE.

100	80.0	80.4	79.4	78.0	83.0	80.4	82.0	79.8	79.0	69.4	68.0	71.2	70.0	69.4	70.2
50	40.0	37.6	39.2	39.2	41.0	41.8	40.2	40.4	39.0	34.0	35.8	35.4	35.4	35.4	35.0
25	19.6	19.4	19.4	20.0	20.0	19.8	19.6	18.6	18.4	18.4	16.8	18.0	18.2	18.8	19.2
10	7.8		7.8	8.0		7.8	8.0		8.0	7.4		7.6	7.5		7.4
5	4.0						4.0			3.6			3.6		
2.5	2.0			2.8			2.0								
0		6.0	1.4		7.8	2.4		6.4	1.4		5.6	0.8		4.8	1.4

SHORT METHOD FOR THE QUANTITATIVE DETERMINATION OF SALICYLIC ACID.

From the results of the qualitative test made by Mr. Ash it appeared that it would be advantageous to confine the work with the quantitative method to carbon tetrachlorid and toluene. Accordingly, Mr. Ash applied the method to various types of wine containing known amounts of salicylic acid varying from 2.5 to 100 mg per 100 cc. One hundred cubic centimeters of the wine were acidified with 5 cc of sulphuric acid (1 to 3) and 50 cc of the solvent were added, gently but thoroughly mixed, and the solvent separated after centrifuging; 25 cc of the solvent were transferred to a weighed watch glass by means of a pipette. With toluene the best results were obtained using a watch glass 4.5 inches in diameter and with carbon tetrachlorid one 4 inches in diameter.

The solvent was allowed to evaporate spontaneously and the amount of residue determined by weighing. The residue was then dissolved in 5 cc of neutral alcohol and transferred into a small casserole, the watch glass being washed thoroughly with neutral boiling water and the salicylic acid titrated with one-hundredth normal barium hydroxid, 1 cc of the reagent being equal to 1.38 mg of salicylic acid.

An aliquot part of the solvent was allowed to evaporate spontaneously, the residue dissolved in 2 or 3 cc of alcohol and diluted with water sufficiently for the colorimetric determination with ferric chlorid. When the amount of salicylic acid present in the original sample was not less than 25 mg per 100 cc, the results obtained by weighing and titration were far superior to those obtained by the colorimetric method, but with smaller amounts the last method was the only one applicable. No tannin was found in any of the residues and the ferric chlorid reactions were clear and entirely characteristic of pure salicylic acid.

In the gravimetric and volumetric determination small amounts of soluble substances were extracted by the solvent. The weight of the residue from 25 cc of the solvent used in extracting normal wine varied from 2 to 3 mg and its acidity was equal to about 0.5 cc of one-hundredth normal barium hydroxid. The results obtained by Mr. Ash on different types of wine are given in the table. In each case these results are the average of three closely agreeing determinations.

It will be noted that the results obtained by Mr. Ash with each solvent and with each type of wine are entirely consistent, and the results obtained by weighing and by titrating the residue agree closely with each other. For instance, by means of carbon tetrachlorid approximately 50 per cent of the salicylic acid present is extracted from dry, red, and white wines. The same reagent, however, extracted slightly more than 60 per cent of the salicylic acid present in the sherry, port, and angelica. Toluene, on the other hand, was found to extract about 70 per cent of the salicylic acid present in dry, white, and red wine and about 80 per cent of that present in sherry, port, and angelica. It would appear that this method might be used advantageously at least for a preliminary determination of the amount of salicylic acid present.

The method was further examined by the referee and by P. B. Dunbar with a view to determining the reason for the varying results obtained with the different types of wine and so modifying the method that uniform results with all substances might be obtained. This investigation was confined entirely to carbon tetrachlorid because of the noninflammability of that substance. Known amounts of salicylic acid were dissolved in dilute alcohol, varying in concentration from 5 to 50 per cent by volume. One hundred cubic centimeters of these dilute solutions of alcohol, containing 1 mg of salicylic acid per cubic centimeter, were shaken in a separatory funnel with 50 cc of carbon tetrachlorid and the amount of salicylic acid determined in 25 cc of the solvent. The figure so obtained was multiplied by two for the purpose of determining the percentage of the salicylic acid extracted in the total amount of solvent employed. The following results were obtained:

Alcohol by volume.	Salicylic acid recovered.	Alcohol by volume.	Salicylic acid recovered.
<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
5.....	44.0	25.....	64.0
10.....	50.0	30.....	64.5
15.....	60.0	40.....	58.6
20.....	61.8	50.....	46.0

It will be noted that the percentage of salicylic acid recovered under these conditions increases with the alcoholic content of the solution up to 25 per cent and then decreases, the maximum results being obtained with from 25 to 30 per cent of alcohol. California red wines, dry and sweet, were then treated in the same manner. Their alcoholic content was increased to 25 per cent and salicylic acid was dissolved in them to the extent of 1 mg per cubic centimeter. Several determinations of salicylic acid were then made by the method described and in each instance 61.2 per cent of the amount of salicylic acid added was recovered. The method was also applied to 100 cc of a solution containing 10 grams of sugar and 25 per cent of alcohol by volume. The amount of salicylic acid recovered from this solution was practically identical with that recovered from a 25 per cent solution of alcohol. Blanks were also run by extracting with carbon tetrachlorid 20, 30, 40, and 50 per cent alcohol acidified with 5 cc of sulphuric acid (1 to 3). It was found that no sulphuric acid was extracted and it was therefore unnecessary to wash the carbon tetrachlorid solution with water after extraction.

DETERMINATION OF SALICYLIC ACID IN DARK BEER AND OTHER CARAMELIZED SUBSTANCES.

Attention has frequently been called to the possibility of error in the determination of salicylic acid in malt extract and beer prepared from highly colored malt and in highly caramelized substances, such as certain varieties of breakfast food.^a This

^a J. Brand, *Zts. gesam. Brauw.*, 1893, 16: 303; H. Kiliani and M. Bazlen, *Berichte*, 1894, 27 (3): 3115-20; *Amer. Brewer's Rev.*, 1907, 21 (5): 222; *Western Brewer*, 1907, 32 (8): 456.

matter was independently studied by A. M. Doyle and P. B. Dunbar, of the Bureau of Chemistry. Both reported that the color given by ferric chlorid with the material extracted from highly colored malt by ether was quite different from the salicylic acid and one should not be mistaken for the other, although the presence of a small amount of salicylic acid may readily be masked by the material extracted from highly colored malt and similar material.

Experiments on malt-nutrine alone and on malt-nutrine containing salicylic acid (100 mg per liter) indicate that there is some possibility of being deceived by the color when ferric chlorid is added directly to the dish containing the dried residue obtained by evaporating the ether solution. In this case a color is sometimes developed which slightly resembles the salicylic acid reaction. If this color is examined in a good light and compared with the color developed by salicylic acid and ferric chlorid there is little danger of being deceived. It is better, however, to carry the evaporation of the extract to about 5 cc on the steam bath and then to complete the evaporation by means of a blast of air, since heating to dryness may darken the residue. The dry residue should be dissolved in a little hot water and ferric chlorid added to this solution. Under these conditions it seems impossible to mistake the salicylic acid reaction. Millon's reagent, freshly prepared, which has been suggested for the detection of salicylic acid in such substances, was not found by either Miss Doyle or Mr. Dunbar to be as satisfactory as ferric chlorid. In the absence of salicylic acid this reagent gives a light pink color, whereas in its presence a deep red is given. The intensity of the color seems to vary so much with the time of boiling, however, as to render the reaction uncertain and unsatisfactory. Both methods were also applied to highly caramelized breakfast foods, to which these observations also apply.

DETERMINATION OF BENZOIC ACID.

Several methods for the quantitative determination of benzoic acid have been suggested recently. Among these the following have been studied by the referee and his collaborators: (1) La Wall's method; (2) La Wall's method modified by mixing a definite weight of tomato ketchup with sufficient saturated sodium chlorid solution to make a definite volume, filtering, and extracting an aliquot portion of the filtrate with chloroform; (3) precipitation as copper benzoate; (4) precipitation as silver benzoate; (5) distillation with steam after decomposing organic matter with sulphuric acid and extracting with ether. It will be noted that the first four methods depend on extracting the benzoic acid from the food or an aliquot extract of the food by means of an immiscible solvent, whereas the fifth depends on separating the preservative from the food by distilling with steam.

Before applying the first four methods a preliminary study was made of the relative advantages of several solvents. This question has been greatly altered by the introduction of the principle of "salting out" the benzoic acid by means of a saturated solution of sodium chlorid. The benzoic acid is thus rendered much less soluble in the solution from which it is extracted and consequently much more readily extracted by the immiscible solvent. The relation of the immiscible solvent to the preservative thus more nearly coincides with the direct solubility of the former in the latter. An approximate determination was therefore made of benzoic acid in several solvents. It was found that the solvents more commonly employed dissolved benzoic acid as follows, 100 cc of the solvent being used in each case: Ether, 25.70 grams; chloroform, 17.34 grams; carbon tetrachlorid, 7.65 grams; toluene, 7.58 grams; benzol, 6.40 grams.

The chief desiderata in the extraction of a substance of this kind are, first, completeness of extraction; second, freedom of the extract from interfering substances; and third, noninflammability. Of the four solvents mentioned, it would appear from the solubility that by far the most complete extraction can be obtained by means of ether, and this is known to be true. Ether is objectionable, however, because of its property of dissolving water and its consequent tendency to extract tannin, salts, min-

eral acids, and other interfering substances. The use of ether is also a source of danger because of its great inflammability.

Chloroform has always been found inapplicable to the extraction of benzoic acid because of the incompleteness of the extraction so effected. Of the solvents studied, however, it stands second to ether in its power of dissolving benzoic acid. A study was therefore made of the efficiency of chloroform in extracting benzoic acid in a saturated sodium chlorid solution, and the extraction was found to be practically complete.

In order to determine the efficiency of chloroform with respect to the extraction of interfering substances, 500 cc of saturated sodium chlorid solution were acidified with 5 cc of sulphuric acid (1 to 5), and extracted with four portions of chloroform containing 100, 50, 50, and 25 cc, respectively. Each portion of chloroform was removed as completely as possible from the solution, the four portions mixed and distilled on a hot plate to a low volume, the last being removed at ordinary temperature in a current of dry air. The residue left by evaporating the chloroform was not weighable. It was dissolved in water and titrated with tenth-normal alkali. Duplicate determinations required 0.03 and 0.04 cc of tenth-normal alkali for neutralization, thus indicating that the error, owing to the extraction of mineral acids under the conditions noted, is not greater than 0.6 mg of sodium benzoate.

One-half gram of tannin was then dissolved in 500 cc of saturated salt solution and extracted with chloroform as just described. The residue left by evaporating the chloroform was not weighable, but required in the duplicate determinations 0.02 and 0.04 cc of tenth-normal alkali for neutralization, which is equivalent to 0.5 mg of sodium benzoate. One-half gram of tannin and 5 cc of sulphuric acid (1 to 5) were then added to 500 cc of saturated sodium chlorid solution and extracted with chloroform as described. The residue obtained by extracting with chloroform was not weighable, duplicate determinations requiring 0.05 and 0.07 cc of alkali, respectively, for their neutralization, equivalent to 0.9 mg of sodium benzoate.

The presence of acetic acid in the ketchups did not appear to cause any inaccuracy. In order to determine whether a large amount of acetic acid would lead to erroneous results, 1 cc of 99.5 per cent acetic acid was dissolved in 200 cc of chloroform and various portions dried, as in the case of the chloroform extract from the samples of ketchup, and the residues titrated. Ten cubic centimeters of the chloroform solution without evaporation require 9 cc of tenth-normal alkali to neutralize it, equivalent to 0.052 gram of acetic acid, or to 0.141 gram of sodium benzoate. Ten cubic centimeter portions of the solution were evaporated before an air blast until no liquid was visible, and 10 cc of water were added to the dish and titrated. An alkaline reaction was secured on the addition of 0.2 cc of one-hundredth-normal alkali. When 50 cc portions were evaporated in the same way and 10 cc of neutral water added to the dish 0.2 cc of one-hundredth-normal alkali were required to give an alkaline reaction to phenolphthalein. When 10 cc portions were evaporated until the chloroform had apparently disappeared but a few drops of liquid remained. The odor of acetic acid was very apparent and the residue required 0.2 cc of tenth-normal alkali for neutralization. Ten cubic centimeter portions were evaporated until the chloroform had apparently disappeared, but still contained a slight amount of liquid with a strong odor of acetic acid. The residue was placed overnight in a sulphuric acid desiccator, 10 cc of water were added to the dish and titrated as above, requiring 0.1 cc of one-hundredth-normal alkali to give an alkaline reaction. It is apparent that while acetic acid will be extracted by the chloroform, no error is occasioned if care be taken to dry the residue either in a current of air or in a desiccator. It was also found that no appreciable error was caused by the presence of tartaric or citric acid. Lactic acid, when present, was extracted to a considerable extent by chloroform, but after being evaporated to dryness before an air-blast the error amounted to only 0.02 per cent.

If the volume of salt solution taken in this work is equivalent to 100 grams of food extracted as in the case of the subsequent studies, the error caused by the extraction of interfering bodies by means of chloroform is less than 0.001 per cent, and is negligible. It would appear, therefore, that chloroform possesses the following advantages: First, after saturating a solution with sodium chlorid a practically complete extraction of benzoic acid may be made; second, the benzoic acid extracted is not accompanied by other bodies that interfere with its determination by means of titration; third, the solvent is not combustible; fourth, it is inexpensive. In connection with subsequent work on the determination of benzoic acid, therefore, no study was made of any other solvent than chloroform except as described under Method IV. The detail of the methods studied is as follows:

LA WALL AND BRADSHAW METHOD (METHOD I).

This method was described in full by the authors in the *American Journal of Pharmacy* (volume 80, pages 171-172). The principle depends upon the method outlined by F. X. Moerk in an article published in the *Proceedings of the Pennsylvania Pharmaceutical Association* for 1905, page 181. The details of the method are as follows:

To 20 grams of the substance under examination add 2 grams of sodium chlorid, 5 cc of hydrochloric acid, and 25 cc of a saturated solution of sodium chlorid. Shake thoroughly for five minutes, transfer to a moistened filter, and wash with a saturated solution of sodium chlorid until 100 cc of the filtrate are collected. Transfer the filtrate to a separatory funnel and shake with three portions of chloroform, using 25, 15, and 10 cc respectively. Evaporate the chloroform at room temperature. If the residue is white and crystalline, dry over sulphuric acid in a desiccator and weigh. If yellowish and oily, dissolve in 10 or 15 cc of weak ammonia acidified with dilute sulphuric acid and again extract with chloroform. The residue is dissolved in 3 to 5 cc of neutral alkali and titrated with twentieth-normal alkali solution, using phenolphthalein as indicator. The titration should agree closely with the gravimetric determination, the difference being rarely more than 1 or 2 mg.

As will be seen by a comparison of the figures obtained by various methods given in the table on page 71, the results by weighing the benzoic acid, given under Method I, are much higher than those obtained by titration. All collaborators report difficulty in securing an adequate filtration. F. W. Heyl obtains very satisfactory results by titration, whereas the figures of all other analysts were low. In the Division of Foods the method was applied to the examination of a considerable number of commercial ketchups. With the low-grade product, made from skin and core pulp, filtration was possible though somewhat slow. With many of the high-grade ketchups the material was so finely divided that filtration was almost impossible. In this connection should be considered the limited solubility of benzoic acid in a saturated solution of sodium chlorid to be mentioned subsequently.

MODIFICATION OF LA WALL AND BRADSHAW METHOD (METHOD II).

This method is identical with the one just described, except that no attempt is made to obtain a complete filtration.

To 200 grams of ketchup are added 20 grams of finely powdered sodium chlorid in a liter flask, and enough of a saturated solution of sodium chlorid is added to make a liter. The contents of the flask are thoroughly mixed and allowed to stand overnight, when they are filtered, and 500 cc of the filtrate are transferred to a separatory funnel, treated with 5 cc of sulphuric acid (1 to 5) and extracted repeatedly with chloroform, using 100, 50, 50, and 25 cc of chloroform, respectively.

In this extraction a troublesome emulsion was formed which it was found could be broken up to a considerable extent by centrifuging and by stirring with a glass rod. Where a centrifuge is not available much may be accomplished by swinging the separatory funnel with the hand. It is important that after each extraction the chloroform be removed as completely as possible, and at the same time the utmost care must be exercised to prevent any emulsion passing through with the chloroform.

Owing to the mineral acid present in the aqueous liquid, the presence of a slight amount of this emulsion in the chloroform layer causes serious error in a titration of the residue.

The results obtained by this method, both by weighing and by titrating the chloroform residue, are given in the table below, under Method II. It will be observed that in all cases the results obtained by weighing are obviously too high, while the results obtained by titration with this method are on the whole very satisfactory. It would appear therefore that the method is fairly satisfactory for the determination of benzoic acid in tomato ketchup, which presumably offers as great difficulties for this determination as any food to which this preservative is commonly added.

Determination of sodium benzoate in ketchup by three methods.

Analyst.	Sodium benzoate added.	Sodium benzoate found.					
		Method I.		Method II.		Precipitation by silver nitrate. ^a	
		By weight.	Titrated.	By weight.	Titrated.		
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	
W. L. Dubois.....	0.247	0.171	0.166	0.232	0.179	0.166	
Do.....	.146	.130	.261	.223	.172	.143	
F. W. Heyl.....	.108	.185	.170	.109	.167		
Do.....	.108	.185	.170	.163	.155		
Do.....	.108	.190	.170				
Do.....	.108	.183	.162				
Do.....	.108	.169	.154				
Do.....	.108	.178	.162				
Do.....	.030						.029
Do.....	.060						.056
Do.....	.060						.056
Do.....	.060						.056
Do.....	.060						.059
P. B. Dunbar.....	.247			.270	.244	.238	
Do.....	.247			.258	.245	.241	
Do.....	.247			.262	.245	.242	
Do.....	.247			.277	.250	.250	
Do.....	.108			.171	.170	.204	
Do.....	.108			.183	.170	.168	
Do.....	.108			.196	.169	.172	
Do.....	.108			.195	.171	.180	
M. C. Albrech.....	.033	.035	.024				
Do.....	.033	.036	.030				
Do.....	.164			.183	.133		
Do.....	.164			.199	.149		
Do.....	.164			b. 177	b. 150		
Do.....	.164			b. 162	b. 150		
C. Conover.....	.060			c. 062			
Do.....	.060			c. 062	.055		
Do.....	.060			c. 063	.055		
Do.....	.060			c. 063	.056		
H. L. Schulz.....	.168	.137	.142			.142	
Do.....	.168	.147	.140				
F. F. Flanders.....	.247	.257	.230			.236	
Do.....	.247	.253				.222	
Do.....	.168	.176	.148			.139	
Do.....	.168	.206					

^a Method IV, pages 74, 75.

^b 100 grams ketchup diluted with water to 200 cc, mixed, and filtered, 50 cc portions of filtrate, diluted to 100 cc, saturated with sodium chlorid, acidified, and extracted with four portions of 100 cc each of ether.

^c Same as footnote "a" except extraction is made with chloroform instead of ether, three portions being used of 100, 50, and 50 cc, respectively.

In some cases where the mixture of tomato ketchup and a saturated salt solution was filtered soon after it was prepared, the amount of benzoic acid determined by this method was too low. This suggests the possibility that the solution of the benzoic acid in the saturated salt solution may not have been complete. More satisfactory results were obtained in all cases where the mixture was allowed to stand overnight before filtering.

An approximate determination of the solubility of benzoic acid in a saturated solution of sodium chlorid was made by P. B. Dunbar. In duplicate tests 0.260 and 0.259 gram of benzoic acid were dissolved in 500 cc of saturated solution of sodium chlorid. In another duplicate determination 0.274 and 0.277 gram of benzoic acid were found to be soluble in 500 grams of saturated solution of sodium chlorid to which 5 grams of sulphuric acid (1 to 5) had been added. No special precautions were taken with respect to the temperature of solutions or the purity of the sodium chlorid employed.

It is apparent therefore that a larger amount of benzoate of soda than 0.27 gram can probably not be determined by this method. The fact that a slightly higher amount is reported in some determinations may probably be explained by slightly higher temperature, or slightly lower concentration of the sodium chlorid solution employed.

It is obviously important that the method be so modified as to permit the determination of a larger amount of benzoate of soda. Attempts were made to extract the benzoic acid from the ketchup by means of water, filtering, and saturating an aliquot part of the filtrate with sodium chlorid. The results obtained were not altogether satisfactory, although the amount of work done was not sufficient to permit of definite conclusions. It is probable that by extracting in this manner or with a saturated sodium chlorid solution, previously made slightly alkaline with sodium hydroxid, the method can be so modified as to permit of its application to samples carrying a much higher percentage of benzoate of soda, and at the same time it will be possible to extract a smaller volume of the salt solution.

PRECIPITATION WITH COPPER BENZOATE (METHOD III).

Mr. C. S. Ash, of the California Wine Association, and Mr. F. W. Liepsner, of the Division of Foods of the Bureau of Chemistry, studied the determination of benzoic acid by means of precipitation as copper benzoate from the residue left by the evaporation of the solvent. It was hoped that this method might afford a means for determining benzoic acid either alone or in the presence of salicylic acid, but preliminary work showed that while copper acetate is a satisfactory precipitant for benzoic acid when present alone, no precipitate is formed in the presence of salicylic acid in considerable quantity, owing probably to the formation of a soluble double salt.

A series of precipitations of benzoic acid were made in alcoholic solutions of various strengths from 0 to 100 per cent and the curve established, giving the per cent of benzoic acid present that may be precipitated as copper benzoate in various strengths of alcohol. The results are as follows:

Precipitation of benzoic acid with varying strengths of alcohol.

Per cent of alcohol.	Per cent of benzoic acid precipitated.	Per cent of alcohol.	Per cent of benzoic acid precipitated.
0	83	50	70
10	84	60	65
20	88	70	58
30	86	80	48
40	75	100	0

It appears that the amount of benzoic acid precipitated is somewhat increased by the presence of alcohol up to 20 per cent of the latter. The increase in strength of the alcohol beyond this point decreases the amount of benzoic acid precipitated, at first slowly and then rapidly, the copper benzoate being completely soluble in strong alcohol. All precipitations were therefore made in a solution containing 20 per cent of alcohol by volume.

To further guard against error due to the solubility of the precipitate, the alcoholic solution from which the precipitation was made was saturated with freshly precipitated basic copper benzoate. A stock solution of the alcohol so saturated was made up and used throughout the work. It was found that the results obtained by precipitation in neutral solution were too high and that a slight acidity in the solution was necessary. This was accomplished by adding a little acetic acid to the copper reagent. The solutions employed and the method of determination were as follows:

Copper acetate reagent.—A mixture of 250 cc of 95 per cent alcohol and 750 cc of water is saturated with copper benzoate and 2 cc of glacial acetic acid added, followed by a concentrated solution of sodium benzoate in 20 per cent alcohol to form a small amount of permanent precipitate. This solution is filtered just before using.

Alcohol solution.—A mixture of 1 part of 95 per cent alcohol and 3 parts of water is saturated with basic copper benzoate, prepared by precipitating copper acetate solution with sodium benzoate, filtering, and washing. An excess of the basic copper benzoate is added to the alcohol in order that additional alcohol may be added from time to time.

Determination.—The residue obtained by the evaporation of the chloroform extract is dissolved in a small amount of the alcohol saturated with copper acetate mentioned above and washed into a small beaker with the same solution, 25 cc of the copper acetate solution is added, the mixture stirred, allowed to stand for one hour, filtered on a gooch, and the precipitate washed with the alcohol solution saturated with basic copper benzoate. The solution is then dried and weighed as usual. If preferred, the residue may be washed into a 100 cc flask with the alcohol solution described, the copper acetate reagent added, and the mixture brought up to mark with the alcohol saturated copper benzoate. The whole is then mixed and, after standing one hour, filtered, and the copper determined volumetrically by any of the standard methods.

The following table shows the results obtained, working on a known amount of benzoic acid in aqueous solution. As before stated above, this precipitate consists of basic copper benzoate, represented by the formula $C_6H_5COO-Cu-OH$.

Benzoic acid recovered from an aqueous solution by Method III.

Benzoic acid present.	Benzoic acid recovered by—	
	Gravimetric method (as copper benzoate).	Titration excess of copper.
<i>Grams.</i>	<i>Per cent.</i>	<i>Per cent.</i>
0.04	83.5	84.1
.04	84.5	75.4
.06	97.9	97.9
.06	100.0	97.9
.08	101.5	
.08	100.5	
.10	101.0	99.1
.10	102.8	104.2
.14	102.0	100.5
.14	102.5	100.5
.20	101.5	103.6
.20	101.8	105.1

An attempt was made to apply this method to the determination of benzoic acid in ketchup. A number of samples were treated as follows:

Three hundred grams were made up to 1,500 cc with saturated salt solution, allowed to stand overnight, and filtered; 500 cc of the filtrate were acidified with sulphuric acid and extracted with 3 portions of chloroform of 100, 50, and 50 cc. The extract was evaporated in a current of air, dried over sulphuric acid, the residue weighed, taken up in neutral alcohol, and titrated with tenth-normal sodium hydrate. The solution was then evaporated to dryness and benzoic acid determined gravimetrically, as described above, by use of the copper precipitation method.

The results obtained by this method are as follows:

Determination of known amounts of benzoic acid in ketchups by Method III.

Sodium benzoate present.	Sodium benzoate found by titration.	Sodium benzoate found by copper precipitation.
Gram.	Gram.	Gram.
0.258	0.229	0.116
.369	.322	.167
.127	.101	No precipitate.
.431	.392	.183
.198	.149	No precipitate.
.387	.328	.174
.107	.079	No precipitate.
.352	.314	.222
.332	.288	.167
.251	.230	.138
.210	.193	.126
.108	.082	No precipitate.
.355	.303	.150
.307	.286	.152
.324	.277	.142
.146	.119	No precipitate.

An examination of this table shows that when there was present less than 0.15 gram of sodium benzoate no precipitate was formed, and in such cases as did give a precipitate all results were from 0.1 to 0.15 gram low. This seemed to indicate that there was something in the ketchup extract which held back the precipitation and showed conclusively that the method could not be used for such materials in combination with present extraction methods.

It appeared probable that since the completeness of precipitation varied with the alcoholic strength it might be interfered with by sugars, higher alcohols, oils from spices, etc., and the following experiments were performed. The alcoholic extract from 1 gram of spice was added to solutions containing 0.1 gram of sodium benzoate and to blanks. Also 0.1 gram of sugar, glycerin, and dextrin were added to similar solutions with the following results:

Determination of sodium benzoate in the presence of spices, sugars, etc., by Method III.

Substance added.	Sodium benzoate present.	Sodium benzoate found.	Substance added.	Sodium benzoate present.	Sodium benzoate found.
	Gram.	Gram.		Gram.	Gram.
Allspice.....	0.1	0.0741	Sugar.....	0.1	0.0941
	.0	.0576		.0	None.
Cinnamon.....	.1	.065	Glycerin.....	.1	.0955
	.0	.0511		.0	None.
Cloves.....	.1	.1621	Dextrin.....	.1	.0854
	.0	.0809		.0	
Black pepper.....	.1	.0634	Check.....	.1	.0977
	.0	.0477			
Red pepper.....	.1	.1293			
	.0	.1478			

These figures show that it is the presence of the spices which causes the failure of this method for the determination of benzoic acid in such products. It is possible that it may be applicable to other materials.

PRECIPITATION AS SILVER BENZOATE (METHOD IV).

This method was suggested and elaborated by Mr. W. E. Hillyer.

The sample is extracted by means of ether as directed in Bulletin 107, page 179, or by the method given by Dubois for the extraction of ketchups.^a The amount

^a J. Amer. Chem. Soc., 1906, 28: 1616.

of substance used should be such that the portion subsequently extracted with ether will contain approximately 0.1 gram of sodium benzoate. The ether extract, after washing with water, is allowed to evaporate to dryness spontaneously, or the first portion of the ether may be distilled and recovered. After drying completely the residue is taken up with a small amount of absolute alcohol, for the purpose of separating interfering substances as far as possible, and filtered into a small beaker. The alcohol is neutralized with sodium hydroxid, evaporated to dryness, and redissolved in a few cubic centimeters of alcohol saturated with silver benzoate. The solution is filtered if not clear, washed with a few drops of aldehyde-free alcohol, saturated with silver benzoate, and treated with from 10 to 15 cc of a saturated solution of silver nitrate in aldehyde-free alcohol. The precipitate is collected in a gooch, care being taken that the asbestos filter be so constructed as to afford as rapid filtration as possible. The precipitate is then heated in a water-jacketed oven until the ether is driven off, cooled, and weighed.

Care must be taken to perform all operations as quickly as possible in order to prevent the separation of silver oxid. The aldehyde-free alcohol mentioned above is about 95 per cent by volume, and is prepared according to the directions given in Bulletin 107, page 96, with the additional precaution of distilling over soda after treatment with meta-phenylene-diamin hydrochlorid. This method involves the use of a considerable quantity of ether, which is objectionable because of its inflammability and the tendency to dissolve sodium chlorid and other interfering substances. Notwithstanding this, very satisfactory results are reported by Messrs. Hillyer and Flanders in the following table. No other results obtained by this method, using ether as solvent, were reported, though the precipitation of benzoic acid as silver acetate, using chloroform as a solvent, was included in the work of several other collaborators.

Determination of benzoic acid as silver benzoate in tomato ketchup (Hillyer and Flanders).

Sodium benzoate added.	Sodium benzoate found.	Sodium benzoate added.	Sodium benzoate found.
<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
0	0	0.060	0.056
0	.004	.060	.056
0	.001	.060	.059
0	0	.146	.101
0	.003	.146	.127
0	.003	.100	.100
0	.001	a. 247	a. 232
.030	.029	a. 247	a. 222
.060	.056	a. 118	a. 159

a Last three results by F. F. Flanders; others by W. E. Hillyer.

These results were obtained by extracting and precipitating as silver benzoate in the ether residue following Method II as given on page 70. The figures seem to be in every way comparable with those obtained by Method II (see p. 71). Extraction with ether appears to be much less satisfactory than extraction with chloroform, owing to the removal of interfering substances by the solvent. These bodies are partially removed by means of absolute alcohol, but this introduces an additional operation and the results obtained are not as satisfactory as by extracting with chloroform from a solution saturated with sodium chlorid. In the table comparing Methods I and II (p. 71), is given the percentage of sodium benzoate precipitated as silver benzoate from the residue from Method II; that is, the liquid titrated under Method II was evaporated to dryness and used as a starting point for the silver benzoate method.

In the following table are given the results obtained by the examination of a number of samples of commercial ketchups using this method. In all cases the benzoic acid was extracted by chloroform from a saturated sodium chlorid solution. Here, again, it will be seen that the results obtained by weighing the residue are in all cases slightly

higher than those given by titration, whereas the amounts determined by precipitation as silver benzoate are almost identical with the amount obtained by titrating the chloroform residue. This method is evidently worthy of further study. It is much more tedious than Method II, but is of value for the purpose of checking the results obtained by that method when a further confirmation seems desirable.

Determination of benzoic acid in commercial samples of tomato ketchup by precipitation as silver salt from chloroform extract.

Analyst.	Chloroform residue weighed.	Chloroform residue titrated.	Silver precipitate.	Analyst.	Chloroform residue weighed.	Chloroform residue titrated.	Silver precipitate.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
C. P. Wilson.....	0.30	0.28	0.28	P. B. Dunbar.....	0.20	0.17	0.18
Do.....	.11	.10	.09	Do.....	.28	.26	.26
Do.....	.19	.16	.15	Do.....	.09	.08	.07
Do.....	.16	.14	.14	Do.....	.21	.19	.18
Do.....	.20	.17	.16	Do.....	.20	.17	.18
Do.....	.33	.32	.31	Do.....	.17	.15	.15

These samples were treated according to Method II, given on page 70. The residue obtained by the evaporation of the chloroform extract was first weighed, then dissolved in about 5 cc of neutral alcohol, the solution so obtained diluted with water and titrated with saturated alkali solution. This solution when exactly neutralized is evaporated to dryness, after which the benzoic acid was determined by precipitation as silver benzoate.

THE DISTILLATION OF BENZOIC ACID FROM SULPHURIC ACID SOLUTION (METHOD V).

This method was suggested and elaborated by Mr. R. M. West,^a and depends on the distillation of benzoic acid with steam after the addition of sufficient concentrated sulphuric acid to insure the complete charring of vegetable tissue and prevent volatilization of coloring matter and oil. The distillation is conducted by means of a flask shown on page 21, the procedure being as follows:

About 10 grams of the sample are weighed into the inner flask of the apparatus, 1.5 to 2 grams of paraffin added, and the flask connected with the condenser. Ten cubic centimeters of strong sulphuric acid are added through a drop funnel at a rate sufficient to complete the addition at from two to three minutes, the flask is gently agitated, to mix the contents thoroughly, and allowed to stand from five to ten minutes after all apparent action of the sulphuric acid has ceased. About 150 cc of distilled water are placed in the outer flask of the apparatus and the water slowly brought to a boil and the boiling continued until 100 cc of the distillate have been collected. The stopcock in the outer flask is left open until the water has heated sufficiently to prevent the contents of the inner flask being drawn into the outer flask.

The distillate is filtered into a separatory funnel and the original receiver and filter are washed with two portions of water of about 10 cc each. The distillate is then extracted with three portions of ether of 50, 30, and 20 cc, respectively. The combined ether extracts are washed repeatedly with water until a 25 cc portion requires not more than 0.10 cc of decinormal alkali for neutralization. The ether extract is then distilled to small volume, after which it is evaporated before a blast of air, dried in a desiccator to constant weight and weighed. The residue is also dissolved in neutral alkali, using phenolphthalein as indicator.

The results obtained by titration agree closely with those obtained by weighing. Excessive foaming is likely to occur when the steam begins to pass into the inner flask. This may be caused by distilling too soon after the addition of the acid, by an insufficient amount of paraffin, or by an unusual amount of sugar in the ketchup. Care must be exercised to prevent the foam passing into the condenser.

^a J. Ind. and Eng. Chem., 1909, 1: 190.

The distillation should be conducted at such a rate that 100 cc of the distillate may be obtained in from twenty-five to thirty-five minutes. Occasionally some paraffin is carried over mechanically, and this may usually be removed from the surface of the distillate by means of a wire or glass rod.

The following results were obtained on ketchups containing a known amount of sodium benzoate:

Determination of sodium benzoate in ketchup by Method V (West).

Sodium benzoate added.	Sodium benzoate found.	
	By weight.	By titration.
Per cent.	Per cent.	Per cent.
0.00	0.01	0.01
.10	.09	.10
.25	.25	.24
.50	.47	.47

THE DETECTION OF CINNAMIC ACID.

The statement has frequently been made that cinnamic acid is being used for the preservation of foods, especially in the case of tomato ketchup. The claim has often been made by those interested in the preservation of ketchup with benzoic acid that the presence of cinnamic acid could not be detected and that firms claiming to use no preservative were preserving with that substance. Two qualitative methods for the detection of cinnamic acid, differing slightly from each other, were elaborated by P. B. Dunbar. Both of these methods depend upon the well-known fact that cinnamic acid is oxidized to benzaldehyde by dilute chromic acid mixture.

Method 1.—One hundred grams of ketchup were treated with 100 cc of water and 5 cc of sulphuric acid (1 to 5) and the mixture extracted directly with three portions of chloroform, using 50, 25, and 25 cc, respectively. The chloroform extract was made alkaline with ammonia and evaporated to dryness on the water bath. The residue was dissolved in a small amount of hot water, filtered, again evaporated to dryness, and heated to boiling with 5 cc of dilute chromic acid mixture (1 part of dilute sulphuric acid saturated with potassium bichromate and 7 parts water). The odor of benzaldehyde is strongest when the mixture is cooled until the fumes of sulphuric acid are no longer apparent.

Method 2.—Two hundred grams of the ketchup are diluted to 500 cc with water, allowed to settle, and filtered. An aliquot portion of the filtrate, 250 cc or more, is acidified with 5 cc of sulphuric acid (1 to 5), extracted with chloroform, and the remainder of the operation conducted as under Method 1.

The second method appears to be slightly more delicate than the first, although with either it was possible to detect cinnamic acid in tomato ketchup when present to an extent of 25 mg per kilogram.

This reaction is also given by cinnamic aldehyde. The method, therefore, does not distinguish of itself between cinnamic aldehyde, resulting from the use of cinnamon as a flavor and cinnamic acid used as a preservative, except that the amount of cinnamic aldehyde present in the commercial ketchups examined was not sufficient to give a reaction. If cinnamic acid were present in the ketchup, it would be detected by the methods used for the detection of benzoic acid. Cinnamic aldehyde, on the other hand, would not be detected by the methods suggested for benzoic acid. The benzoic-acid residue obtained by the evaporation of the chloroform extract may be examined by the cinnamic-acid methods described.

The germicidal and antiseptic properties of cinnamic acid were investigated by G. W. Stiles, who found them to be very much lower than those of benzoic acid. The preservation of a food, therefore, would require a much larger percentage of cinnamic

acid than benzoic acid. In fact, the antiseptic properties of a saturated solution of cinnamic acid are so slight that this substance would probably not serve as a preservative for foods.

A method for the separation of benzoic acid and cinnamic acid by precipitation of the latter with manganous salts^a was tried unsuccessfully by Mr. Dunbar, who was unable to secure a precipitation of either benzoate or cinnamate of manganese in dilute solution. As is to be expected, Mohler's and Peter's reaction also give the same end reaction in the presence of cinnamic acid.

REPORT ON TEA, COFFEE, AND COCOA.

By A. G. WOODMAN, *Associate Referee.*

The work of the referee for the past year has been limited to a study of methods for the determination of caffeine and cafetannic acid in coffee, extract in tea, crude fiber and starch in chocolate, and sugars in milk chocolate. Twenty-two samples were prepared and sent out to those who had expressed a willingness to collaborate, ten on tea and coffee and twelve on cocoa products. These were accompanied by the following directions and a letter of transmittal:

CAFFETANNIC ACID.

(a) *Krug's method.*—Proceed as directed in Bul. 107, p. 155. (Note that the formula for lead cafetannate should be $Pb_2(C_{15}H_{15}O_8)_2$, as in Bul. 107, Rev.) Save the filtrate for the determination of caffeine. After weighing the lead cafetannate determine its lead content as follows: Digest with aqua regia, add sulphuric acid, heat to fumes, cool, dilute, add alcohol, settle, filter, ignite, and weigh as lead sulphate. Calculate as per cent of lead.

(b) *Method of Trillisch and Göckel.*^b—Boil 3 grams of coffee one-half hour with water, filter, and repeat this treatment on the residue three times. The united filtrates are made up to 1,000 cc. To 400 cc add 1 cc of basic lead acetate solution and allow to stand overnight. Filter, wash, decompose the precipitate with sulphuretted hydrogen, filter from lead sulphid, evaporate to dryness, and weigh.

CAFFEIN.

In the filtrate from the lead cafetannate precipitate the lead with hydrogen sulphid, filter, and remove the excess of hydrogen sulphid by boiling, concentrating the solution, if necessary, to about 100 to 150 cc. Add tenth-normal potassium iodid solution of iodine in excess, filter through a little glass wool and determine the excess of iodine with tenth-normal sodium thiosulphate.

1 cc tenth-normal iodine equals 0.00485 gram caffeine.^c

EXTRACT IN TEA.

(a) Follow the provisional method as described in Bul. 107, p. 149.

(b) Follow the method proposed by Doolittle and Woodruff (Bul. 105, p. 48).

CRUDE FIBER (SAMPLE A).

Proceed as directed in Bul. 107 under "VI. General Methods," 11, page 56, except that the fiber is filtered and weighed on a paper. The sample should be pulverized by grinding with ether as described in the succeeding paragraph.

CRUDE STARCH (COPPER-REDUCING MATTERS BY DIRECT ACID HYDROLYSIS), SAMPLE A.

Weigh 4 grams of the material if unsweetened, or 10 grams if sweetened, into a small wedgewood mortar, add 25 cc of ether and grind with a pestle. After the coarser material has settled decant off the ether together with the fine suspended matter on

^a Scoville, Amer. J. Pharm., 1907, 79 [12]: 549-551.

^b Zts. Nahr. Genussm., 1898, 101.

^c Gomberg: J. Amer. Chem. Soc., 1896, 18: 331.

a 11 cm, blue ribbon, S. and S. paper. Repeat this treatment until no more coarse material remains. After the ether has evaporated from the filter, transfer the fat-free residue to the mortar by means of a jet of cold water and rub to an even paste, filtering on the paper previously employed. Repeat this process until all sugar is removed. In the case of sweetened products the filtrate should measure at least 500 cc. Conduct the hydrolysis of the residue as directed for "Starch" under "VI. General Methods," 8 (a), page 53, Bul. 107, Rev., except that after neutralizing with sodium hydroxide, add 5 cc of basic lead acetate solution (prepared as directed under "VI. General Methods," 6 (b), (1), page 40) before completing the volume to 250 cc. To 100 cc of the filtrate add 1 cc of 60 per cent sulphuric acid, filter off the lead sulphate and determine reducing matters in 25 cc of the filtrate as directed under "VI. General Methods," for Reducing Sugars, 7, (b), (2), page 49. Determine copper by the direct weighing of cuprous oxide, 7, (c), (6), page 53.

SUGARS (SAMPLE B).

Determine the lactose and sucrose as described by Dubois.^a

The amount of work requested was purposely made small in order that it should not prove burdensome, but in spite of this results were received from only four chemists, two on cocoa products and two on tea and coffee.

TEA AND COFFEE.

The results on tea and coffee are shown in the following table:

Cooperative work on coffee and tea.

Analyst.	Caffetannic acid.		Caffeine.	Extract in tea.	
	Krug method.	Trillich and Göckel method.		Krauch method.	Doolittle and Woodruff method.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
F. O. Woodruff, New York.....	12.65	14.42	0.38		52.95
	12.41	17.10	.33		51.50
	11.05	15.26	.29	45.64	51.69
	12.17	16.26	.53	(Whole)	50.92
	11.25	14.28	.38	40.74	51.42
	11.49	16.09		(Ground)	45.90
	12.27				
	12.23				
	12.27				
	12.28				
A. G. Woodman, Boston.....	11.19	8.03	.68	39.60	49.72
	13.21	7.80	.75	42.15	50.16
		8.26	.88	40.82	50.28
		8.22	.74		50.99
			.78		
Mark Millikin, Hamilton, Ohio.....			.81		
	11.09				
	11.86	9.33	.67		44.8
	10.11	8.8	1.06		42.7

COMMENT BY MR. WOODRUFF.

Krug method for caffetannic acid.—(1) The water must be kept to constant volume during thirty-six hours' digestion.

(2) Unless great care is used, the addition of lead acetate to the hot alcohol solution will cause violent ebullition and partial loss of contents. A safety tube helps to overcome this difficulty.

(3) In determining the lead content of the caffetannate it is advisable to filter the caffetannate through a tared gooch. This will allow of digestion of contents in nitric acid and precipitation of the lead with sulphuric acid without using a filter paper, the carbon of which does not completely oxidize and produces a blackening of the lead sulphate. The final weighing of the sulphate should also be made in a gooch.

^a J. Amer. Chem. Soc., 1907, 29: 560.

Caffein method.—It is suggested that the caffein iodine precipitate does not form immediately and that the low results are due to filtering and titrating the solution too quickly. Other work indicates that after the iodine is added the flask should be allowed to stand in an ice chest overnight before titrating.

Much better results can be secured by Gomberg's original method for caffein, as given in the *Journal of the American Chemical Society* (1896, 18: 331), and modified as follows:

Extract 2 grams some time with four portions of water, cool, and make to 1,000 cc. Treat 500 cc with 15 cc of saturated lead acetate solution, let settle, filter, remove lead with hydrogen sulphid, boil off excess of hydrogen sulphid, divide filtrate into two parts, concentrate each to 50 cc, add 0.2 cc of concentrated hydrochloric acid to one and 0.5 cc of acetic acid to the other, cool to 15° C., add 20 cc of tenth-normal iodine solution, stopper flask, and let stand in ice two hours, filter on a gooch. Caffein does not precipitate unless mineral acid is present, so the acetic acid portion shows if any other materials are present which would precipitate with the iodine solution. If any absorption of iodine is found in the acetic portion, it must be deducted from the titration containing mineral acid. The difference represents the iodine used up in the formation of the periodide of caffein: 1 cc of tenth-normal iodine equals 0.00485 gram of caffein. Using this method, 0.78 per cent of caffein was obtained from the coffee reported.

Krauch method for extract in tea.—The bulk of sample (20 grams) makes complete removal of water-soluble substances almost impossible. The absorption of water by large filter paper and on surface of flask during weighing is also a serious objection to the method. If sample is ground, filter paper is clogged and filtration prevented.

Doolittle and Woodruff method.—Care should be used to keep the entire sample in the boiling liquid during extraction or low results will be obtained. Any loss of water by evaporation should be replaced.

NOTES BY REFEREE.

The discrepancy in the results obtained by the two analysts with the Trillich and Göckel method is due principally to the fact that Mr. Woodruff used 5 cc of basic lead acetate in the precipitation instead of 1 cc, as prescribed in the method. Determinations made by the referee on the same sample gave 10.08 per cent where 2 cc of basic lead acetate was used and 12.04 per cent when 4 cc was used. The lower results obtained by Mr. Woodruff in the caffein estimation may have been due to the greater volume of solution in which the caffein periodide was precipitated, he using a volume of 100 to 150 cc, while the referee employed a volume of 20 cc. Experiments made by Mr. W. C. Taylor in the writer's laboratory have shown the necessity for concentrating the caffein solution to small bulk.

The determinations made of extract in tea by the referee convinced him of the great superiority of the Doolittle and Woodruff modification over the Krauch method as regards convenience, time, and liability to error.

COCOA PRODUCTS.

The following results were obtained from the collaborating chemists on cocoa products:

Cooperative work on cocoa products.

Analyst.	Plain chocolate (Sample A).		Milk chocolate (Sample B).	
	Crude fiber.	Crude starch.	Lactose.	Sucrose.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
G. M. Bartlett, Boston.....	2.25	11.61	5.05	25.72
R. W. Hiltz, Philadelphia.....	2.50	9.69	4.83	27.83
	2.65	9.97	5.25	27.32
A. G. Woodman, Boston.....	2.84	10.37	5.13	27.02
	2.69	10.03		

G. M. Bartlett: The conversion of the starch was carried out as outlined, no difficulty occurring in the procedure. The aliquot for precipitation was obtained as follows: After converting, neutralizing, and adding basic lead acetate the sample was made up to volume at about 35° C. To 100 cc at this temperature was added the 60 per cent of sulphuric acid, cooled so that the volume of liquid contracted to 100 cc. It was necessary to cool only to about 18° C. The sample for precipitation was taken when the liquid had contracted to the mark. Two determinations were made—one by precipitating by the Walker-Munson method (*J. Amer. Chem. Soc.*, June, 1906,) and the other following the method in Bulletin 107. The latter gave 11.83 per cent of starch.

In determining crude fiber the electric stove was used for boiling the 1.25 per cent sulphuric acid and caustic soda. There was but little frothing. The filter paper and crude fiber were dried at 100° C and over sulphuric acid. During weighing the filter paper gained in weight. I do not care for this method of getting the weight of the crude fiber, even though it is not to be ignited, and would prefer filtering on a weighed platinum gooch filter.

In calculating the sugar in chocolate by Dubois's method ^a it seems illogical to multiply $(a-b)$ by $1.05x$ (x equaling the volume obtained by dissolving sugar in 100 cc of water) rather than by 105 plus the increase in volume due to the solution of the sugar. This actually makes but little difference in the result, but the following statement of the formula seems preferable:

$$\frac{(a-b)(105+x')}{144-\frac{t}{2}} = \text{per cent sucrose.}$$

Where x' = increase of volume owing to the solution of the sugar in water. In calculating the lactose the complete formula reads: Per cent lactose = $C \times 4 \times 1.11 \times 1.05x \times 1.264$, where x = volume of solution when the sugar is dissolved in 100 cc.

R. W. Hilt: The samples on arrival were immediately placed in glass-stoppered bottles. Before removing portions for analysis they were rubbed down to a coarse powder in a large porcelain mortar and mixed as well as possible. This was done quite rapidly, both to avoid possible changes in moisture content and to avoid formation of a pasty mass.

Crude fiber: First filtration was made on closely woven linen in a 4-inch Büchner funnel with light suction. Second filtration was on a 11 cm B & A ashless filter paper without suction. Both filtrations were rapid and satisfactory.

Starch: Results are multiplied by the factor 1.01 to correct for the dilution of 100 cc of the solution by the 1 cc of sulphuric acid.

Sugars: The method of Dubois was followed exactly. It was necessary in extracting with water to break up with a glass rod the compact cake left after centrifuging the last time with gasoline. Inversions were made in the cold (50 cc + 5 cc of hydrochloric acid, being allowed to stand over night. All volumes were adjusted at 20° and all polarizations were made in jacketed tube at exact temperatures. The actual polariscope readings (averages of four to five close readings) illustrate the very great influence that small differences in readings have upon the results, in these dilute solutions. In spite of this fact, the method seems to be satisfactory and convenient for judging milk chocolates. The methods are, in my opinion, in as simple a form as possible, and can not well be improved.

RECOMMENDATIONS.

In view of the small number of collaborators it is hardly possible for the referee to make any formal recommendations based on collaborative work. It is evident, however, that the study of certain of these methods should be continued by the association, especially the caffeine estimation and the determination of sugars in chocolate. There would appear to be no reason why the determination of extract in tea as outlined by Doolittle and Woodruff should not be substituted for the cumbersome Krauch method. The experience of the referee on numerous samples of cocoa products suggests that the requirement for filtering and weighing the crude fiber on a

^a *J. Amer. Chem. Soc.*, 1907, 29: 556; see also Bul. 107, Rev., p. 256.

paper should be omitted, as the determination can be made more conveniently on a gooch crucible as ordinarily used.

Attention is also called to the accompanying paper involving some of Mr. W. C. Taylor's work on caffetannic acid and caffein.

ESTIMATION OF CAFFETANNIC ACID AND CAFFEIN IN COFFEE.

By A. G. WOODMAN and W. C. TAYLOR.

In connection with an examination of the methods for coffee analysis the writers have made a study, in the limited time available, of the provisional methods for determining caffetannic acid and caffein, especially the former.

CAFFETANNIC ACID.

Experience has shown that with the directions as given at present it is practically impossible to obtain concordant results or a lead caffetannate of constant composition.

It has been the general experience of those who have worked with the Krug method that it is tedious in the extreme, and, furthermore, that the composition of the so-called lead caffetannate obtained varies with the conditions of precipitation. It was our purpose to ascertain if possible the source of some of these difficulties.

It was seen early in the work that variations in the amount of lead acetate used for precipitation gave variations in the proportion of lead caffetannate obtained, as well as in its content of lead. This is shown in the following table, in which the determinations were made on aliquot portions of a coffee infusion and varying amounts of saturated lead acetate were used, all other conditions being kept constant.

Determination of caffetannic acid, using varying amounts of lead acetate.

Lead acetate.	Caffetannic acid by Krug's factor.	Lead in precipitate.
cc.	Per cent.	Per cent.
1	7.66	50.04
2	9.73	50.35
4	11.14	50.35
6	11.70	48.31
8	12.28	55.16
10	14.28	55.93

The averages of several results are stated in each case, although the results showed very considerable variation. While too much reliance can not be placed on these figures, owing to variations among themselves, they show the necessity of using a definite amount of lead acetate for the precipitation.

Another source of error is the difficulty of washing the lead caffetannate free from lead acetate. Those who have attempted it know the tediousness and the difficulty of washing the precipitate on the filter. It is of course necessary to use alcohol of 90 per cent strength in washing, on account of the solubility of the precipitate in water or dilute alcohol. On the other hand, the lead acetate which is to be removed is only slightly soluble in 90 per cent alcohol. Hence it will be readily seen that it is practically impossible to wash the bulky precipitate on the filter. It is true, also, that when the wash water no longer reacts for lead the precipitate is not necessarily free from it, since owing to its character the wash water easily forms channels and does not wash it thoroughly.

After numerous experiments, washing by the centrifugal machine was tried, giving several treatments with 90 per cent alcohol in the tubes of the centrifugal before trans-

ferring to filter paper. This method gave results which were in much closer agreement, as shown by the following results on the same sample:

Per cent caffetannic acid.....	9.69	9.69	9.57	9.40
Per cent lead.....	48.28	48.35	48.01	48.71

Tests made on a considerable quantity of the lead caffetannate washed in this way showed it to be free from fat and nitrogen.

It would seem as if the long process of digestion with water and with alcohol prescribed by Krug could be materially shortened. In much of our work extracts of the coffee were prepared by the use of a shaking machine, shaking the sample for an hour with water and half an hour with alcohol. Results obtained in this way agree very well with those obtained by the official method of digestion, although there is evidence to show that neither method extracts all of the caffetannic acid.

Regarding the vexed question of the composition of caffetannic acid, we seem not much nearer a settlement. The views previously held, which seem to lead to the formula for a di-glucosid, have been clearly set forth in Bulletin 105 by Mr. Howard. Lack of time prevented any extended investigation of this problem, but an endeavor was made to confirm the work of Cazeneuve and Haddon in regard to the di-glucosid formula for caffetannic acid. We were unsuccessful, however, in preparing more than traces of the osazone prepared by them, although carrying out the experiments exactly in the manner prescribed. In this connection the paper recently published by Görter^a is of interest, in which the correctness of the Cazeneuve and Haddon formula is questioned. Görter states that he was unable to form more than a few small crystals of the osazone, which he was unable to isolate and considered that it was due to some impurity in the caffetannic acid. The caffetannic acid is considered by Görter to be a mixture of chlorogenic and coffalic acids. Numerous derivatives and salts of these acids have been prepared and are described by the author to support his contention.

The method for carrying out the Krug test which we found to work most satisfactorily may be summed up as follows:

To 2 grams finely ground coffee (passing 0.5 mm sieve), add 10 cc of water and shake for an hour in a mechanical shaking device. Add 25 cc of 90 per cent alcohol and shake again for half an hour. Filter and wash with 90 per cent alcohol. Bring the united filtrate and washings, about 50 cc, to boiling and add 6 cc of saturated lead acetate solution. Separate the precipitated lead caffetannate by means of a centrifuge, decanting the supernatant liquid through a tared filter. Repeat the centrifugal treatment twice with 90 per cent alcohol, decanting each time through the filter. Transfer the precipitate to the filter and wash free from lead. Wash with ether, dry at 100°, and weigh. The weight of precipitate multiplied by 0.51597 gives the weight of caffetannic acid.

CAFFEIN.

In the work on caffein a comparison was made of three methods: The official method (Bul. 107, p. 154); the titration of caffein with iodine, according to Gomberg, in the filtrate from the lead caffetannate; and the method proposed by Görter in the paper previously mentioned.

Our attention has been directed by Mr. C. D. Howard to a source of error in the provisional method, arising from the fact that the extraction with dry chloroform of the sand-magnesia mixture does not yield the whole of the caffein. Mr. Howard says in his letter:

My practice has been to add to the concentrated filtrate, contained in a tin-foil dish, about 10 grams of sand and 1 gram of magnesium oxid, evaporate and dry in the water oven for a short time. The brittle mass, easily stripped from the dish, I grind finely, place in a paper extraction cartridge, and extract for ten to twelve hours in the usual way.

^a Annalen, 1908, 359: 217.

Now, my recent experience has been that if the extracted residue be shaken with water and the latter further extracted, an additional quantity of caffeine, sometimes equivalent to 10 per cent of the whole, is thus obtained.

Our experience has been a similar one. To illustrate by a specific instance, the residue from twenty hours' extraction with chloroform was shaken with water, filtered, and the aqueous solution extracted four times with chloroform. One-half the chloroform extract was tested for caffeine, and gave positive tests with Wagner's reagent and by the "murexid" test. The other portion showed by a Kjeldahl determination 0.0035 gram of caffeine, corresponding on the whole sample to about 10 per cent of the amount present.

Görter finds that a considerable proportion of the caffeine in coffee is present as a double salt, the potassium caffeine chlorogenate, from which the caffeine is extracted by dry chloroform only with great difficulty. Whether or not this be the cause of the incomplete extraction, it is evident that the official method needs revision.

Preliminary experiments with the Gomberg method showed it to be practicable for the small amount of caffeine (approximately 20 mg) that would be present in the filtrate from the lead caffetannate, providing the volume of solution were not over 25 cc. On account of the slight solubility of the caffeine periodid in the wash water it was found best in working with this small amount to suck the precipitate as dry as possible on the gooch filter and not to wash it. Determinations made on 20 mg of caffeine in 25 cc of water in this way gave from 98 to 99 per cent of the caffeine present. Numerous experiments made on the filtrate from the lead caffetannate precipitate by precipitating the lead with hydrogen sulphid and evaporating the filtrate gave fairly concordant results, which were uniformly lower than those given on the same coffee by the other methods for caffeine. It was observed that the variations in amount of caffeine as determined in this way corresponded roughly with the variations in amount of caffetannic acid as found by the Krug method. Whether these variations and low results are due to incomplete extraction of caffeine by the process of digestion employed in the Krug method is a matter which we expect to investigate further.

Görter's method was not given a thorough trial. As far as the work goes it has been satisfactory, and the method is worthy of further trial by the association. It reads briefly as follows:

Eleven grams of the finely powdered coffee are moistened with 3 cc of water and after standing a half hour extracted for three hours in a Soxhlet extractor with chloroform. The extract is evaporated, the residue of fat and caffeine treated with hot water, filtered through a cotton plug, and washed with hot water. The filtrate and washings are made up to 55 cc, 50 cc pipetted off and extracted four times with chloroform. This chloroform extract is evaporated in a tared flask and the caffeine dried at 100° and weighed.

In the determinations made it has never been possible to weigh the caffeine directly on account of impurities, the caffeine having been calculated from a determination of nitrogen in each case. From the work done there seems to be a strong probability that a combination of the Gomberg and Görter methods will prove to be the best and most convenient process for determining caffeine in coffee.

SECOND DAY.

FRIDAY—MORNING SESSION.

REPORT ON THE DETERMINATION OF NITROGEN.

By CHARLES L. PENNY, *Referee.*

An apology is due the association for failure to carry out the instructions of 1906 concerning the permanganate methods. Another phase of the nitrogen question considered last year and discussed in correspondence with the National Fertilizer Association seemed to outweigh in importance and urgency all others, namely, the determination of total nitrogen in mixed fertilizers to which nitrate of soda is added. With a view to making a thorough investigation of this subject, the following instructions were sent to such members of this association as were supposed to be interested in the work and to more than a score of analysts named by the secretary of the National Fertilizer Association:

INSTRUCTIONS.

Chemists cooperating in this work are requested to give their attention exclusively to methods applicable in the presence of nitrates, Bulletin 107, page 7 (c), page 8 (d), page 10 (g) and (h). There seems to be urgent need of this investigation, especially since the present methods used to determine nitrate nitrogen have been formally called in question by the representatives of great commercial interests.

No samples are sent herewith, as it is thought that each analyst, by calculating the nitrate used from a solution of nitric acid carefully compared with his own standard alkali and acid, may get more reliable results, through the balancing of possible errors, than from using a common substance.

Let the source of nitrate used be a solution of pure nitric acid, about fifth normal, most accurately titrated against the standard alkali used in the Kjeldahl work. In each case measure accurately into the digestion flask enough of this nitric acid to contain 30 to 50 milligrams of nitrogen.

(1) Follow method (c), disregarding the water in the nitric acid and without neutralizing.

(2) Follow likewise method (d).

(3) Follow method (g), adjusting the proper amount of water, distilling first with magnesia, then with caustic soda and water added to the residue in the distillation flask, collecting *separate* distillates and titrating each *separately*.

(4) Similarly follow method (h), beginning in second line "in a distillation flask," etc.

If the yield of nitrogen is less than the calculated in (1), (2), (3), or (4), test the residue in distillation flask for nitrates.

(5) Proceed as in (1), (2), (3), (4) with the preliminary addition of 2 grams of cane sugar to the digestion flask.

(6) Proceed as in (1), (2), (3), (4) with the preliminary addition of 1 gram, accurately weighed, of organic nitrogen substance, such as dried blood, fish scrap, or tankage, to the digestion flask. In a separate operation treat 1 gram, accurately weighed, of this added substance similarly except that no nitrate be present; that is, analyze the added substance alone according to (c), (d), (g), and (h). Any nitrate-free mixed fertilizer may be used for the added substance.

While the above plan entails much work, it is hoped that a large number of chemists will test at least some of the several official methods in question, if not all. The figures for each separate determination should be reported and the precise method pursued should be fully explained. The results of any other plan of studying the nitrate question, carried out by chemists at their own suggestion, will be welcomed.

Answers were received from five chemists engaged in official work and three engaged in commercial work, viz, Messrs. E. M. Bailey, New Haven, Conn.; F. B. Carpenter, Richmond, Va., reporting work of Mr. W. D. Cooke; H. S. Lansdale, Buffalo, N. Y.; C. B. Morrison, New Haven, Conn.; J. Bernard Robb, Richmond, Va.; B. F. Robertson, Clemson College, S. C.; Paul Rudnick, Chicago, Ill., and T. C. Trescot, Washington, D. C.

It is regretted that cooperation has not been more general, but the work required seemed burdensome, and doubtless few could find the time to engage in it. Several of the cooperating chemists, however, have shown extraordinary industry, reporting an amount of work seldom equaled in voluntary investigation of this sort. The questions involved in the plan of work are not less than 14; hence analytical results are too complicated to admit of convenient tabulation. It seems better, therefore, to deduce from the analytical figures the answers to the several questions.

Before judging the results it is well to bear in mind the reasonable expectation of agreement or accuracy from a number of chemists working on the same subject. Last year on the simpler problem of determining nitrate-free nitrogen, the work of over 50 chemists, possibly the largest number of the association ever engaged on a single question at one time, seems to indicate that about 98 per cent of the truth is the average with present methods and present personnel. Then in the more difficult question of nitrates and the separation of several forms of nitrogen, this expectation would seem to be at least high enough. Thus methods for nitrates that give as much as 98 per cent of theory are at least as accurate as the average results on nitrate-free substance. While this limit may easily be exceeded by experienced and skillful individual analysts, it is useless to deny that it is not exceeded by the average results.

The analysts used chiefly as their source of nitrate nitrogen amounts of their own standardized nitric acid containing from 28 to 160 mgs of nitrogen. The results obtained are reported as percentages, the basis of which is the amount of nitrogen that should have been obtained.

The questions involved follow, with the answers deduced from the figures of the several analysts.

Percentages of nitrogen recorded based on amount present, using different methods.

[Is nitric acid, in the absence of organic matter, reduced to ammonia without loss?]

(1) BY METHOD (C).

Analyst.	Number of determinations.	Range of determinations.	Average.
		<i>Per cent.</i>	<i>Per cent.</i>
Bailey.....	2	74.5-82.9	78.7
Cooke.....	5	94.0-96.0	95.0
Lansdale.....	2	97.4-98.6	98.0
Morrison.....	2	86.6-86.7	86.7
Robb.....	8	87.0-96.0	91.4
Robertson.....	4		99.2
Rudnick ^a (Thio.).....	4	61.1-76.9	67.4
(Zn).....	4	80.8-86.8	83.2
(Zn + Thio.).....	4	76.4-82.5	79.8

(2) BY METHOD (D).

Bailey.....	2	74.8- 75.7	75.3
Cooke.....	3	96.0- 96.0	96.0
Lansdale.....	2	95.7- 97.4	96.6
Morrison.....	1		76.4
Robb.....	3	93.0- 95.0	94.0
Robertson.....	4		98.5
Rudnick.....	8	55.0-100.0	76.0

^a Analytical work reported by F. W. Rudnick throughout the report was done by F. Fenger, K. J. Monrad, and A. C. Johnson.

Percentages of nitrogen recorded based on amount present, using different methods—Cont'd.

(3) BY METHOD (G).

[Distilled off magnesia followed by soda, the sum of both distillates being used.]

Analyst.	Number of determinations.	Range of determinations.	Average.
		<i>Per cent.</i>	<i>Per cent.</i>
Bailey.....	2	99.8-100.0	99.9
Cooke.....	2	94.0-95.0	94.5
Morrison.....	2	98.1-98.9	98.5
Robb.....	6	89.0-98.0	93.5
Rudnick.....	8	84.9-95.0	76.8

(4) BY METHOD (H).

Bailey.....	2	95.6-99.3	97.5
Morrison.....	2	95.4-97.2	96.3
Rudnick.....	12	91.6-99.9	95.8

[Is nitric acid reduced to ammonia without loss in the presence of 1.4 to 2 grams of sugar?]

(5) BY METHOD (C).

Bailey.....	2	35.6-49.5	42.6
Morrison.....	2	39.6-63.9	51.8
Robb.....	3	61.0-63.0	61.7
Robertson.....	4	-----	100.0
Rudnick.....	8	71.7-86.9	79.4
Robertson ^a	4	-----	98.0

(6) BY METHOD (D).

Bailey.....	2	40.0-42.1	41.1
Morrison.....	2	24.8-44.2	34.5
Robb.....	3	61.0-63.0	61.7
Robertson.....	4	-----	99.5
Rudnick.....	8	70.0-86.2	76.7
Robertson ^a	4	-----	99.0

(7) BY METHOD (G).

[Distilled off magnesia followed by soda, the sum of both distillates being used.]

Bailey.....	2	95.4-98.9	97.2
Morrison.....	2	98.0-99.3	98.7
Rudnick.....	3	103.6-111.2	^b 106.6

(8) BY METHOD (H).

Bailey.....	2	97.0-110.0	103.5
Morrison.....	2	95.8-98.1	97.0

[Is the sum of nitrogen in nitric acid and nitrogenous organic matter fully recovered as ammonia?]

(9) BY METHOD (C).

Bailey.....	2	79.2-86.5	82.9
Morrison.....	2	84.0-87.1	85.6
Rudnick (Thio.).....	3	77.4-82.4	80.7
(Zn+Thio.).....	4	66.6-74.7	70.6

^a In the presence of 1 gram of a nitrate-free mixed fertilizer instead of sugar.^b In these determinations by the Ulsch method, the small quantity of water undoubtedly caused spurting of alkali sufficient to drive it over into the condenser. The bumping was terrific. Rudnick.

Percentages of nitrogen recorded based on amount present, using different methods—Cont'd.

(10) BY METHOD (D).^a

Analyst.	Number of determinations.	Range of determinations.	Average.
		<i>Per cent.</i>	<i>Per cent.</i>
Bailey.....	2	77.5-81.0	79.3
Morrison.....	2	77.3-86.5	81.9
Rudnick.....	8	42.6-43.7	55.0

[Is any of the nitrogen in nitrate-free and ammonia-free nitrogenous bodies obtained?]

(11) BY METHOD (G).^a

Bailey.....	2	1.0-2.4	2.2
Morrison.....	2	2.4-3.5	3.0

^a These figures were obtained distilling with magnesia only. Soda being added and distillation continued, the following additional percentages were obtained: Bailey, 8.3 to 9.3, average 8.8; Morrison, 8.3 to 11, average 9.7. These four determinations were made on cotton-seed meal containing 0.0739 gram of nitrogen, on which nitrogen the percentages are based.

(12) BY METHOD (F).

Bailey and Morrison, who alone worked on this question, report that distillation was rendered impossible by excessive frothing.

[How complete is the liberation of ammonia when distilled with magnesia?]

(13) BY METHOD (D).^a

Analyst.	Number of determinations.	Range of determinations.	Average.
		<i>Per cent.</i>	<i>Per cent.</i>
Bailey.....	4	92.2-97.9	94.3
Cooke.....	2	19.0-22.3	20.7
Morrison.....	4	93.6-97.9	95.8
Robb.....	6	15.5-31.6	22.4
Rudnick.....	8	42.4-73.7	63.8

^a These figures represent the fraction of total ammonia liberated by magnesia, the complement lacking to make 100 per cent having been liberated by soda. Thus, Cooke's lowest result was 19 per cent distilled from magnesia and the remaining 18 per cent was obtained by adding soda solution and continuing the distillation. Trescot, reported below, recovered practically all of the ammonia by magnesia alone.

(14) Is the loss of nitrogen by methods (c) and (d) caused by heat generated in mixing the acid and water?

Rudnick, using potassium nitrate instead of a solution of nitric acid, obtained the following figures, and as compared with these results those found with the solution of nitric acid by the same methods averaged 79.8 to 66.1 per cent. Trescot obtained 80 per cent when using nitric acid, as compared with 100 per cent with potassium nitrate.

Rudnick's results, using potassium nitrate.

Method.	Number of determinations.	Range of determinations.	Average.
		<i>Per cent.</i>	<i>Per cent.</i>
(c) (Zn+Thio.)..	4	93.5-97.4	95.4
(d).....	4	88.3-94.0	92.1

The work of Trescot on potassium nitrate is reported separately, as follows:

(15) BY METHOD (D).

Material.	Number of determinations.	Range of determinations.	Average.
		<i>Per cent.</i>	<i>Per cent.</i>
Potassium nitrate.....	4	99.8-100.1	100.0
Nitric acid, neutralized and evaporated.....	5	97.7- 99.2	98.8
Potassium nitrate and dried blood.....	3	98.0- 98.8	98.5
Potassium nitrate and ground bone.....	3	100.6-101.6	101.1

(16) BY METHOD (G). (ULSCH-STREET.)

Potassium nitrate.....	3	99.8-99.8	99.8
Potassium nitrate and dried blood.....	2	99.2-99.2	99.2
Potassium nitrate and ground bone.....	2	99.2-99.2	99.2

Impurities in reagents, reckoned on the nitrogen as the basis of percentage, were reported as follows by two analysts only:

Percentage of impurities in reagents based on nitrogen present.

Analyst.	Method.			
	(c)	(d)	(g)	(h)
Bailey.....	0.9	0.7	0.4	4.4
Morrison.....	.8	.7	.4	4.4

COMMENT BY ANALYSTS.

Carpenter, reporting the work of Cooke, says:

The Kjeldahl method, or in fact any of the methods described in the "Official Methods of Analysis," are very unsatisfactory for the determination of nitrates. In our own work for the analysis of nitrate of soda, nitrate of potash, etc., we use an electrolytic method.

Robb says:

I noticed a very perceptible loss of nitric oxid when I added the salicylic-acid mixture both in the case of the nitric-acid solutions containing a nitrate-free fertilizer and those containing cane sugar.

Rudnick says:

Inasmuch as all of the work could not be carried out in full, such features as seemed to me of less interest from the standpoint of a fertilizer manufacturer's laboratory were omitted. These had to do chiefly with the determination of nitrate nitrogen per se. We are not so much interested in this feature, inasmuch as there are several good methods now in use for this purpose. Of these we prefer the Schloesing-Wagner method, as described in Bulletin 107 of the Bureau of Chemistry, page 111.

It is not the determination of nitrate nitrogen that we are so vitally interested in but the determination of total nitrogen where nitrates are present.

The results with the fifth-normal nitric acid were very unsatisfactory indeed. This was probably due to the great dilution with water, which we never meet with in our work. Every precaution was taken to prevent undue heating on adding the strong salicyl-sulphonic acid.

A great many more determinations than appear in the preceding report were made in this laboratory, but none of them was any more satisfactory than those reported on, and there seemed to be no special significance in the results obtained. The Ulsch method as given in Bulletin 107 with a number of variations has been tried in this laboratory at various times during the past years, but has never given satisfactory

results. The zinc-iron method works very smoothly, and some of the results are quite as satisfactory as those obtained by the Schloesing-Wagner method.

But, as stated above, we are mostly interested in the Gunning and Kjeldahl methods for total nitrogen in the presence of nitrates. These methods have not given satisfactory results in the past. There are some details in the manipulation of these methods not described in Bulletin 107 which seem to tend materially to a closer approximation of the true amount of nitrate nitrogen, such as quick covering of the sample with enough salicyl-sulphonic acid to absorb all of the evolved nitrogen oxides, sufficient time for thorough nitration of the acid, thorough cooling of the mixture before adding the thiosulphate, and heating to a free boil before adding the mercuric oxid or the potassium sulphate, respectively. We find also that the tendency to foam is very largely obviated by heating the flask containing the mixture in a boiling water bath for an hour, or possibly less, prior to adding the thiosulphate.

In view of the poor results obtained with nitric acid it seemed that the use of a pure nitrate weighed directly into the digestion flask might prove much more satisfactory for determining the accuracy of the various methods. Our work following this plan has given much better results than by using nitric acid as the source of the nitrate.

In the large majority of cases we have to do with mixed fertilizers containing a nitrate. Any inaccuracies due to the water present in nitric acid or in the solution of a nitrate could easily be obviated by evaporating to dryness in the digestion flask, preferably in vacuo, after neutralizing when necessary to prevent the loss of nitric acid.

But even in the determination of an added nitrate the results leave much to be desired. This is not only the case in this particular work, but has been a generally recognized fact among fertilizer chemists for a long time. If the secret of success lies in certain details of manipulation not commonly known, which make the method reliable, these details should certainly be mentioned in the description of the method.

Trescot says:

I inclose results on neutralizing with soda and evaporating down, also on mixtures of potassium nitrate and blood, and potassium nitrate and bone by the Gunning modified method for nitrates and the Ulsch-Street method. These results only confirm my previous work on such materials with these methods. The zinc-iron method is a failure in my hands. I never could get concordant results. If there is anything more I can do, let me know and I will be glad to do it; only I do not wish to repeat my work on the Ulsch-Street and Gunning modified methods, for after the most careful checking for many years I am convinced that if handled properly, and on dry materials, both methods will give all the nitrate present.

DISCUSSION OF RESULTS.

The opinions quoted and results reported show wide variations. By methods (c) or (d) with and without organic matter, Robertson and Trescot alone get satisfactory results; those of the other chemists are low, mostly impossible, in fact. This may be due to heat generated by the acid and water, as many chemists think and as Rudnick's and Trescot's work on potassium nitrate seems to show. On the other hand, Robertson's work shows no appreciable loss. An important question is, Must not this possible loss from heat be reckoned with even with a comparatively dry substance? Even with potassium nitrate Rudnick falls far short of acceptable accuracy. In view of these facts may not a too speedy application of heat in methods (c) and (d) cause loss of nitrate vapors? It is a practice of some careful analysts to allow nitrate samples to stand several hours after the thiosulphate or zinc dust is added. Hence, recommendations are offered fixing a minimum time limit in methods (c) and (d).

Method (g) as carried out by Bailey, Morrison, and Trescot gives good results; as carried out by the other analysts uncertain and varying results, mostly far too low. It must be noted, however, that to test the completeness of the reduction of nitric acid to ammonia the distillation is made at first off magnesia and completed off soda, except in Trescot's work, the amount of ammonia from each distillation being estimated separately and the sum taken. It appears further from this process that the distillation from magnesia is usually far from complete, giving in several instances less than one-fifth of the total amount of ammonia; that is, less than one-fifth of what

should have been obtained. As method (g) permits the use of magnesia only, no soda, in distilling, it is evident how very inaccurately this method is practiced by some chemists. It would seem that the magnesia distillation process is conducted by many chemists less successfully than any other analytical process.

Furthermore, the work of Bailey and Morrison shows that by method (g) some ammonia is obtained from cotton-seed meal, about 2 or 3 per cent of the total nitrogen in the substance coming off as ammonia, when the distillation is off magnesia, and about three times as much in addition, when soda is used in the distillation. This is fortunately a compensating error and contributes something toward making up the deficit just described due to distilling ammonia off magnesia.

As practiced by some chemists the process of distilling off magnesia is worse than useless. As method (g) does not affect the amount of total nitrogen, errors are not so serious, however, as in methods (c) and (d).

Method (h) shows better agreement among different chemists and also gives figures somewhat approximating theory.

The only report on method (f) is to the effect that excessive frothing prevented distillation.

Of the 54 answers to the 14 questions stated above, 39, or nearly three-fourths, must be considered unfavorable, either as failing to reach the 98 per cent standard of accuracy or as showing reactions contrary to the plan and purpose of the method.

It must be admitted, whatever may be the inherent accuracy of the several methods here discussed, that most of them in the hands of some experienced chemists fail to give reasonably reliable and accurate results. It must also be admitted that these methods with more or less variation in detail are commonly accepted by chemists as reliable, and, as this report shows, may be made to give exceptionally accurate results. Obviously methods so firmly established, and by some analysts so successfully employed, are hardly to be condemned, or even seriously questioned, without a study of them by a large number of chemists. The data obtainable for this report are too meager to justify any criticism or proposed radical change.

The referee is still of the opinion that the methods here discussed are fairly accurate when properly followed, less accurate perhaps in the hands of some analysts than methods applied to simpler determinations, such as of nitrate-free nitrogen, but yet as accurate as the difficulty of the case permits; and furthermore, that unquestionably these methods are not always successfully followed, as evidenced by the criticisms of chemists as well as by the reported results, and that probably some analyses made according to these methods are erroneous, giving too low results; that the complaint of the officers of the National Fertilizer Association may possibly be based on fact in certain cases, probably due in part to erroneous analyses and in part to actual loss of nitric acid, but that it is not within the power of this association at present to remedy the evil complained of, if it exists.

RECOMMENDATIONS.

Two recommendations of 1907, referred to the referee for 1908, are recommended for adoption as official. (Nos. 2 and 4, Circular 38, page 1, or Bulletin 116, page 129.) These changes relate to the use of copper sulphate in the Kjeldahl and Gunning methods. For detailed statement of changes see page 183.

Recommendation 3: Bulletin 107 Rev., page 8, fourth line from top, after the word "time" insert: "Allow the flask to stand without heat for not less than six hours."

Recommendation 4: Same reference, page 8, under (d) (3) "determination," fifth line of paragraph, after word "and" insert: "Allow the flask to stand without heat for not less than six hours; then". So changed the sentence beginning with "Add 5 grams" would read: "Add 5 grams of sodium thiosulphate and allow the flask to stand without heat for not less than six hours; then heat the solution for five minutes; cool; add 10 grams," etc.

At the close of the reading of the nitrogen report, the president announced the following committees:

Committee on amendments to the constitution: J. P. Street, J. T. Willard, P. F. Trowbridge.

Committee on nominations: R. J. Davidson, C. H. Jones, B. B. Ross.

Committee on resolutions: L. L. Van Slyke, A. J. Patten, V. K. Chestnut.

REPORT ON INORGANIC PLANT CONSTITUENTS.

By H. D. HASKINS, *Referee.*

The work on inorganic plant constituents has been along lines recommended by the referee of the preceding year, particularly with reference to the development of a method for the determination of iron and aluminum in ash. The sample which has served for the work was prepared by thoroughly mixing the ash of a species of *Lycopodium*, known to contain a large proportion of aluminum, with a finely ground and incinerated sample of wood ashes, the latter being known to contain considerable quantities of iron.

PROPOSED METHODS.

The method proposed for study contains some of the features incorporated in the official method of determining ferric and aluminic oxides and phosphates in soils (Bul. 107, p. 15). See also Bul. 56, Proceedings of the Fifteenth Annual Convention of the association in which recommendations are made by Hartwell in regard to a method described in Crooke's Select Methods of Chemical Analysis. The method as outlined for the work this year was in detail as follows, a hydrochloric acid solution of the ash being used:

SEPARATION OF FERRIC AND ALUMINIC OXIDS IN ASH ANALYSIS.

Use a solution corresponding to 0.2 gram of ash. After removing the phosphoric acid the filtrate from the precipitate of ammonium phosphomolybdate, consisting of a nitric acid solution of molybdic acid, ferric oxid, alumina, lime, and magnesia, is placed in a beaker and cautiously neutralized with ammonia, care being taken that the temperature does not rise above 40° C. and that the alkali is added only in slight excess; allow to stand in a warm place until the precipitate completely settles, filter the clear supernatant fluid, wash the precipitate with hot water by decantation, then transfer it to the filter, and finish the washing. Next, redissolve the precipitate through the filter in weak, hot nitric acid (1 to 5), reprecipitate with ammonia, filter, and wash in the same careful manner. The precipitate is dried, ignited, and weighed as ferric oxid and alumina.

METHOD (b).—The weighed precipitate of ferric oxid and alumina is dissolved, on the hot water bath, in a covered flask by the addition of about 20 cc of dilute sulphuric acid (1 part sulphuric acid to 4 parts water). The iron is reduced to the ferrous state by adding iron-free metallic zinc (about 5 decigrams at each addition) until the solution is completely decolorized and the iron is all reduced; cool by immersing in cold water, dilute with cold distilled water which has been recently boiled, pour off and wash into beaker, leaving behind any residue of zinc. Titrate with standard permanganate solution.

METHOD (c).—An aliquot part of the original solution A, corresponding to 0.2 gram of ash, is evaporated on hot water bath with the addition of 10 cc of sulphuric acid until all hydrochloric acid is expelled; dilute with water, reduce with zinc, and estimate iron by standard solution of potassium permanganate. The per cent of ferric oxid obtained is deducted from the per cent of ferric oxid and alumina, corrections being made for filter ash, to obtain the per cent of alumina.

MAKING AND STANDARDIZING PERMANGANATE SOLUTION.

Dissolve 2.82 grams of pure crystallized potassium permanganate in distilled water by the aid of heat; cool and dilute to 1 liter and preserve in stoppered flask. Standardize this solution by titration with metallic iron solution as directed in the second American edition of Fresenius's Quantitative Chemical Analysis, pages 268-269.

A copy of the method as outlined above, together with the ash sample, was sent to eight chemists who had signified their intention of cooperating in this work. The results received from five analysts are given in the tabulated statement which follows:

Cooperative work on ash sample.

Analyst.	Method B.		Method C.	
	Alumina (Al_2O_3).	Ferric oxid (Fe_2O_3).	Alumina (Al_2O_3).	Ferric oxid (Fe_2O_3).
J. A. Le Clerc.....	6.56	2.54	6.43	2.67
Andrew J. Patten.....	7.12	2.73	7.17	2.68
S. C. Dinsmore.....	^a 7.45	^a 3.35	^a 7.45	^a 3.35
O. M. Shedd.....	6.74	2.66	6.74	2.66
H. D. Haskins.....	6.64	2.63	6.69	2.58
Average.....	6.77	2.64	6.76	2.65

^a Not included in average.

COMMENTS BY ANALYSTS.

J. A. Le Clerc found that it was necessary to redigest the ash residue with hydrochloric acid in order to remove all of the iron present. In Method C some organic matter seemed to interfere with the end point of permanganate titration.

Andrew J. Patten used an approximately hundredth-normal solution of permanganate solution, this being preferred on account of the small amount of iron present.

O. M. Shedd found objection to the determination of iron by Method B in that it takes a long time to dissolve the oxides in the sulphuric acid solution, and at this point recommended the fusion of the oxides with potassium hydrogen sulphate.

The referee is of the opinion that a weaker solution of permanganate is preferable to the one recommended.

The average results obtained by the two methods agree very closely and indicate but little choice in method of procedure.

The variations obtained between the various chemists in the results reported may be due to the method of standardizing the permanganate solution. The referee has used for this purpose a solution made from iron of a known composition furnished by the Bureau of Standards, Washington, D. C. The personal equation must also enter into cooperative work of this nature to a greater or less extent.

RECOMMENDATIONS.

In view of the fact that the two methods gave results agreeing within 0.01 of a per cent, the referee feels justified in recommending the one which involves the least manipulation. The following is therefore recommended as an official method for the separation of iron and aluminum in inorganic plant constituents:

Use an aliquot part of solution A corresponding to 0.2 to 0.5 gram of ash for the determination. After removing the phosphoric acid, place the filtrate from the precipitate of phosphomolybdate, consisting of the nitric acid solution of molybdic acid, ferric oxid, alumina, lime, and magnesia, in a beaker and cautiously neutralize with ammonia, care being taken that the temperature does not rise above 40° C., and that the alkali is added only in slight excess; allow to stand in a warm place until the precipitate completely settles. Filter the clear supernatant fluid, wash the precipitate a couple

of times with hot water by decantation before transferring it to the filter, wash four or five times on the filter with hot water. Dissolve the precipitate through filter with weak, hot nitric acid (1 to 5), reprecipitate with ammonia, filter and wash in the same careful manner. Dry, ignite, and weigh the precipitate as ferric and aluminic oxides.

Transfer an aliquot part of the original solution A, corresponding to 0.2 to 0.5 gram of ash, to an Erlenmeyer flask and evaporate with 10 cc of sulphuric acid on a hot water or steam bath until all of the hydrochloric acid is expelled; dilute with distilled water to original volume and reduce the iron to the ferrous state by adding iron-free metallic zinc (about 5 decigrams at each addition) until the solution is completely decolorized and the iron is all reduced. Cool and estimate the iron by standard solution of potassium permanganate. Deduct the per cent of ferric oxid obtained from the per cent of ferric and aluminic oxides to obtain the per cent of alumina. Use a fiftieth-normal solution of potassium permanganate standardized by a solution of metallic iron of known composition.

It is also recommended that further work be done with the peroxid method for the determination of total sulphur in plants and plant products, as suggested by the committee at the last association meeting. Lack of time has prevented the referee from taking up this important subject during the past year.

REPORT ON MEDICINAL PLANTS AND DRUGS.

By L. F. KEBLER, *Referee*.

Much activity has been shown during the past year by both federal and state officials charged with the enforcement of the various laws governing medicinal agents. The drugs studied were both imported and domestic. Many interesting results have been observed, a few of which will be noted in the referee's report. The feature standing out most prominently is the lack of standards and recognized methods for detecting the presence and determining the amounts of many active medicinal agents, and from the nature of some of these agents no satisfactory methods will probably be provided in the near future. Even some of the methods available and the standards set are found wanting. In this connection it is desirable to call attention to certain features of the Pharmacopœia. A plant product is described, and in some cases a standard relative to alkaloidal content is prescribed, but in many instances no provisions are made for excluding or permitting the presence of any foreign materials, such as stems, sticks, etc. It is a very common experience to meet with importations of leaves containing large quantities of these impurities. The same holds true with other commodities, such as cubeb berries, in which frequently a large percentage of stems and twigs is found, together with unripe or overripe berries, and the question arises, To what extent is it permissible for these materials to be present? It is contended by producers and importers that a standard precluding the presence of these foreign agents would be purely theoretical, academic, if you please, and has no standing in the business world. On the other hand, it is well known that the medicinal value of a preparation is enhanced or depreciated in value in proportion to the quantity of these foreign agents present. For example, the per cent of alkaloid material present in belladonna root will be lowered in proportion, other things being equal, to the amount of adulterant present. In other words, it is depreciated at least in medicinal value proportionately to the foreign material present, and to what extent these foreign bodies unfavorably influence the medicinal action of a drug in which they are found is unknown.

Another feature is the amount of sand or incidental earthy matter present. For example, normal senna leaves do not contain to exceed 10 per cent of sand and other inorganic material, but it is not uncommon to meet with siftings, sweepings, etc., of senna containing from 20 to 35 per cent of such impurities. There is no provision in the Pharmacopœia setting an ash limit to a product of this character, but it is reasonable to expect that sand does not constitute a material part of a normal product used

for medicinal purposes. How many would be willing to administer to children compound licorice powder prepared with senna leaves containing 25 per cent of sand?

Probably no other drug has caused so much annoyance and dissatisfaction during the past year as *asafetida*. When the drug sections of the federal law were put into effect at the various ports it was found that this commodity was brought in containing various amounts of alcohol-soluble material. No important quantity of exceedingly inferior material was offered, and for the time being no detentions were made, even though this drug was somewhat below the strength prescribed by the U.S.P. for alcohol-soluble material. It soon developed, however, that importers were bringing in successive lower grades of this product; for example, the alcohol-soluble material diminished gradually from 40 to 30 to 25 and to 20 per cent, and one consignment was offered containing, according to the declaration on the containers, as low as 15 per cent of this material, while one case of this consignment was found to possess only a trifle over 6 per cent of such alcohol-soluble material. The Pharmacopœia also prescribes an ash limit of 15 per cent. The virtue of *asafetida* resides largely, if not exclusively, in the alcohol-soluble material, and it would therefore seem that the ash limit should be liberal. If an importation were offered containing on the average 50 per cent or more of *asafetida* alcohol-soluble material, but the ash was materially above the limit prescribed by the Pharmacopœia, such an importation should not be considered illegal.

Attention is also directed to another drug, namely, *copaiba*. During the past year large quantities of this product were imported and correspondingly large examinations were made at the ports, particularly New York. It will be recalled that the test as originally prescribed by the committee of revision of the U.S.P. was modified at the earnest solicitation of many dealers in this commodity. The result is that the new method is so unreliable and unsatisfactory as to permit the entry of *copaiba* containing at least 25 per cent of *Gurjun* balsam, the common agent used for its adulteration. A question arising in connection with this commodity is, Shall the definition given relative to *copaiba* as contained in the U.S.P. be strictly adhered to, or shall we recognize under the name *copaiba*, qualified or otherwise, any other commodity which is derived from other sources than those definitely prescribed by the above authority? For example, there is constantly offered for importation a product known as African *copaiba*, which is derived from an entirely different geographical source than the commodity recognized by the Pharmacopœia. It is well known that the African oleoresin differs materially in composition and therapeutic action from the *copaiba* recognized by the U.S.P. African *copaiba* certainly is not *copaiba* within the meaning of the pharmacopœial definition for this commodity.

Another problem requiring attention is the dilution of certain drugs with inert substances. The Pharmacopœia prescribes a lower limit of alkaloidal content for certain potent drugs and it has developed that millers are adding to alkaloidal drugs assaying above the lower limit prescribed by the Pharmacopœia such inert material as powdered olive stones so as to reduce the alkaloidal content to the lowest limit prescribed by the above authority. There is nothing in the Pharmacopœia that would indicate that such a practice was contemplated or recognized. The committee undoubtedly was familiar with the fact that alkaloidal drugs frequently contain a larger amount of alkaloidal material than the lower limit prescribed, but only in one case, and that is opium, is there a specific provision made for the addition of a foreign inert material so as to reduce the product to a strength conforming to both a lower and an upper limit. This is an important question and requires adjustment in the near future.

Attention has also been directed to the fact that there are many commodities on the market which probably owe their virtues to their alcoholic content; for example, there are a number of so-called (for want of better names) medicinal wines, bitters, etc., which contain only a dash of some medicinal substance such as extract of cin-

chona, gentiana, etc., or very small amounts of one or more of the cinchona alkaloids. Mixtures of quinin and whisky are cases in hand. One of the products examined was found to contain not more than one-fortieth of a grain of total alkaloidal matter to an ounce of the product, and even then the alkaloidal matter consisted only in part of quinin. Without informing a prospective consumer or partaker, he, as a rule, would not detect any abnormal odor or taste, excepting the one imparted by the so-called whisky itself. One of the arguments frequently used to justify the existence of products of this character is that the National Formulary, a standard quoted by the food and drugs act, recognizes preparations of a similar type. The preparation referred to most frequently is beef, wine, and iron. The point raised is an exceedingly important one and requires adjustment. If it is permissible to add simply enough of an agent to merely suggest a certain physiological action, be it ever so remote, primarily for the purpose of using the name of a substance possessing recognized medicinal properties, in conjunction with the trade name of a commodity, one helpful feature of the law will be largely negated and an increased number of so-called medicinal products of the most absurd character can be placed upon the market.

As before stated, there are many drugs for which we have no satisfactory chemical methods for determining whether or not given samples are active or inert. In some few cases it has been possible to employ animal experimentation. The drugs amenable to this form of study are digitalis, cannabis indica, strophanthus, etc. The methods at present available appear to give fairly satisfactory results in the hands of experienced operators, but there is much to be done before it can be definitely stated whether or not a given consignment possesses sufficient medicinal properties. One specific case in this connection should be noted, namely, digitalis leaves. This product is of such great importance to the medical practitioner for the treatment of certain heart affections that nothing relative to this drug and its preparation should be left to chance. No less an authority than Doctor Dixon, of England, says: "For my part I unhesitatingly express the belief that many hundreds of patients die annually from digitalis and allies not possessing the virtues which are required of them."

At the meeting of the association last year the referee urged the appointment of a number of associate referees for the purpose of studying certain features which need careful investigation; for example, it is absolutely necessary to have an intimate acquaintance with the macroscopical and microscopical features of crude plant products. Satisfactory methods are also wanting for analyzing the many mixtures containing modern synthetic chemicals, used for the treatment of headache and numerous other affections. We are in possession of fairly reliable methods for determining and estimating morphin in certain combinations or if present in considerable amounts, but it is quite another matter to detect and estimate this agent when present in very minute quantities in mixtures containing various solvents, and much study will be required before this single item is placed upon a footing which will be absolutely reliable. During the past year some work has been attempted along this line with cocain. It may at first appear to be a very simple matter to detect the presence and estimate the amount of cocain in mixtures, but when it is remembered that there are quite a number of products which respond to one or more of the tests laid down for detecting cocain the difficulties can readily be appreciated. Careful, thoroughgoing investigations on all these points are greatly needed, and until more work is done it will be difficult to satisfactorily enforce some features of the federal and state laws dealing with these products.

State officials are making frequent requests for information as to methods of analysis and standards for certain products. The referee has been actively engaged on both of these lines of work and has now in preparation standards for certain products for which no standards of a satisfactory character exist, and in cases where the existing standard is somewhat deficient it is the intention to add certain features which will

enable all workers throughout the United States engaged in the investigation of these products to arrive at just conclusions relative to their quality. For example, it is intended to fix an upper limit of the amount of foreign material that may be present in a leaf described by the United States Pharmacopœia and to provide an ash limit for certain drugs. Considerable progress has also been made relative to testing existing analytical methods and formulating new methods for examining certain commodities.

In order to facilitate the investigation, and in harmony with the instructions of the association, the referee appointed several associates to take up specific features of the work, whose results will be given in separate papers.

A PRELIMINARY STUDY OF THE MICROCHEMICAL ANALYSIS AND IDENTIFICATION OF ALKALOIDS.

By B. J. HOWARD and C. H. STEPHENSON.

The large number of alkaloids used at present in drugs as well as the increasing number of synthetic products being placed on the market has made felt the want of additional means for their identification. Microchemical methods have been suggested and used to a limited extent by such workers as Wormley, Barth, and Behrens, but the principal application of the method has been rather for the purpose of localizing the alkaloid in the tissues, by such investigators as Errera, Maistriau, Clautriau, Bolling, and others.

At the suggestion of the Chief of the Division of Drugs this investigation was taken up by the Microchemical Laboratory and a study of several alkaloids begun. Only a preliminary report of progress can as yet be made, and the field has extended itself in many directions as the work progressed. The investigation as originally outlined anticipated several lines of work, among which the following might be mentioned:

- (1) The normal reaction of each alkaloid with each of the various reagents which are known to be of service with one or more of them. This involves a study of dilution, the dilution of the alkaloid which will respond to the test and also the weight limit of alkaloid which will give positive tests with the reagent. It also involves a study of the forms of crystals produced at the various dilutions, the conditions for producing the reaction, or the manipulation, the determination of melting points, photographing the crystals as a matter of record, and in some cases the measurement of crystal angles.

- (2) A study of the influence upon the reaction of another alkaloid present than the one sought.

- (3) A study of the influence upon these reactions of such substances as glycerin, sugar, starch, oils, and fats, gums, waxes, and other compounds likely to be found in drugs, and from traces of which it is often difficult to remove, for testing, small traces of alkaloids in some medicinal preparations.

- (4) The adaptation of alkaloidal purification methods for use microchemically so as to permit minute quantities of the alkaloids to be separated and prepared for testing.

- (5) The developing of an analytical scheme for systematically identifying microchemically the various alkaloids present in unknown mixtures. This last can only be accomplished after a considerable number have been studied and compared.

During the last year the work has been practically confined to the first two lines, which naturally constitute the foundation of the whole investigation. The alkaloids studied comprise a list of about forty, besides two or three salts of two of them, most of which were obtained through the Division of Drugs. They were commercial specimens and apparently of average purity. The list also embraces several synthetic compounds as well as the more common natural alkaloids. Among the natural alkaloids or their salts studied might be mentioned the following: Cocain, codein, atropin, cinchonin, morphin, papaverin, narcein, caffein, strychnin, tropacocain, hydrastin, coniin, berberin, solanin, etc., while among the synthetic bodies studied are anes-

thesin, beta-eucain, holocain, gujasanol, and acoin. The dilution of the alkaloid in the solvent in many cases has a most marked effect upon the form assumed by the precipitate. There is always a limit beyond which the dilution of the product is too great for crystallization to take place, while on the other hand the concentration may be so great as to cause too sudden precipitation and an unsatisfactory product results. In this work dilutions of 1:100 or 1:200 were most frequently tested. Other dilutions would possibly have given crystalline products where only noncrystalline products have thus far been obtained or where no reaction at all has been noted.

The reagents used embraced a list of more than ninety compounds or mixtures and included the standard reagents and as far as known, the special alkaloidal reagents with the exception of two or three which have recently been brought to the authors' attention. Thus far crystalline precipitates have been obtained in about 400 combinations. Noncrystalline deposits resulted in nearly 600 other combinations, but their usefulness in identification is very limited and they can usually only be employed as corroborative tests.

Unfortunately some of the well known alkaloidal reagents, though giving reactions with most of the alkaloids, produce only noncrystalline precipitates. As ordinary analytical tests they may be satisfactory, but as microchemical reagents they leave much to be desired. To the analytical chemist they serve a good purpose as indicating alkaloidal presence, but rarely its identity. This is shown in the following examples: Mayer's reagent, 11 crystalline, 23 noncrystalline; Kraut's reagent 10 and 33 and Marme's reagent 11 and 25, respectively.

Picralonic acid gave 21 crystalline precipitates out of 37 positive reactions, but the forms unfortunately are in most cases too much alike to be of much service for identification. The alkaloids studied showed a great diversity in the character of the precipitate formed, as is seen from the following examples, which serve to illustrate the extremes, the first four giving a high number of crystalline forms, the last five giving a high number of noncrystalline.

Character of precipitate obtained with different alkaloids.

Alkaloid.	Crystalline.	Noncrystalline.	Alkaloid.	Crystalline.	Noncrystalline.
Strychnin.....	36	3	Apomorphin.....	6	35
Berberin.....	25	6	Papaverin.....	9	32
Tropacocain.....	22	2	Hydrastin.....	1	32
Anæsthesin.....	20	7	Solanin.....	1	29
Acoïn powder.....	2	40			

With piperin, sanguinarin, emetin, and apocodein noncrystalline precipitates only have been obtained thus far, though it may be that by some change of manipulation crystals may yet be produced.

The melting point of the products is likely to be of service at times in establishing the identity of certain compounds though some of the precipitates apparently are too unstable for this test. For this purpose, however, an apparatus which had been devised in the Bureau of Chemistry for use on the stage of the microscope has been tested with promising results. It allows of the microscopic examination and determination of the melting point of an individual crystal in a mixture of various kinds either with plain or polarized light. In some crystals, especially some of the compact spherical forms, this last point is an important means of telling where melting begins, since as soon as a crystal melts it loses its polariscopic activity, and as all systems, except those belonging to the regular system, are active this feature can be used to advantage in determining the point where melting begins and where it ends even on small crystals.

The alkaloid thus far most thoroughly examined is cocain. Crystalline deposits have been obtained with each of the following eleven reagents, viz, palladous chlorid, platinum chlorid, gold chlorid, picric acid, chromic acid and hydrochloric acid, potassium dichromate and hydrochloric acid, potassium permanganate, potassium chromate, sodium carbonate, ferric chlorid, and potassium hydrate or sodium hydrate; noncrystalline deposits were obtained with chlorzinc iodid, picralonic acid, Mayer's reagent, phosphomolybdic acid, phosphotungstic acid, Kraut's reagent, Wagner's reagent, barium mercuric iodid, and potassium cyanid.

The following observations were noted concerning the various reactions with cocain in which crystals were produced:

Palladous chlorid.—This is one of the most characteristic tests for cocain, though not quite so sensitive as gold chlorid. The crystals vary in form greatly, according to the conditions of precipitation. There is at first formed, except in very dilute solutions (1:300 and up), an orange-colored amorphous-like or oily precipitate from which, on standing, crystalline forms of golden brown color are produced. One of the most common forms is that obtained with a 1:100 dilution, when feathery crystals are formed which have a strong tendency to twin. With a solution of 1:20 a dense precipitate is thrown down, out of which hexagonal plates are at first formed and frequently followed later by sheaf-like clusters of fine-pointed acicular crystals. A dilution greater than 1:500 gives crystals only with difficulty, crystallization being induced by rubbing the slide with the glass stirring rod. The limit of the test is 0.2 μ gr.

Platinum chlorid.—With a 1:20 solution a dense white precipitate is formed and quickly followed by the production of very narrow feathery crystals—many times twinned so as to resemble a bird with outspread wings. Clusters of more than two are also abundant. If the reagents are mixed slowly the crystals are more like those of greater dilution. With a 1:100 dilution the feather type is much more prominent, the secondary branches being well developed into frost-like forms. With 1:1,000 solutions either short thick crystals are formed or else plate crystals twinning in a most characteristic manner are produced. The dilution limit is about 1:4,000, and the limit in 1:1,000 is 0.2 μ gr.

Gold chlorid.—This is the most sensitive reagent for cocain so far found. At 1:100 feathery frost-like crystals are produced, together with some nearly smooth star-like aggregates. At 1:1,000 the form is much the same, but the branches usually bear a rough outline. Diamond plates are also produced. At 1:4,000 a cross-like form predominates, the cross-bar being short. A few rosette crystals frequently are present. Crystals can be obtained in dilution up to 1:20,000 and the limit of the test for dilutions of about 1:3,000 is 0.033 μ gr.

Picric acid.—This is a good reagent for dilutions up to 1:800, though the crystals produced are not very characteristic for this alkaloid. They are produced in spherical rosettes (or sheafs) of fine lemon-yellow acicular forms. The reaction takes place quickly, and no difficulty is experienced in producing them nearly to the limit of dilution. At 1:300 the limit is 0.2 μ gr.

Potassium permanganate.—With cocain, solutions up to a dilution of 1:700 give purple-colored square plates, or aggregates of this form. Vigorous rubbing of the slide is often necessary to start the crystallization, which then proceeds readily. When they begin to crystallize spontaneously, the plates are sometimes deposited in spherical aggregates. The limit at 1:400 is 2 μ gr.

Chromic acid and hydrochloric acid.—This test is made by adding a small drop of 5 per cent chromic acid solution to the test drop. A precipitate is formed which on stirring disappears (if too much has not been added). A small drop of strong hydrochloric acid is added and a yellowish deposit is produced, which after rubbing of the slide should in a few moments be transformed into loose spherical clusters of an acicular crystal. This test appears to be one of the most uncertain because of the difficulty with which the crystallization is sometimes induced to begin in dilutions greater than 1:60. A concentration of 1:1,000 has produced positive results on standing several minutes. The limit appears to be for 1:100 about 3 μ gr.

Potassium bichromate and hydrochloric acid.—This test gives the same form of crystals as the chromic acid and the test is conducted in a similar manner. The limit of dilution is about 1:1,000 while at 1:100 the limit is 3 μ gr.

Ferric chlorid.—The crystals are spherical aggregates of rather coarse blade-like crystals with chisel-shaped ends. The limit of dilution is about 1:1,000 and in a dilution of 1:100 the limit is 3 μ gr.

Potassium hydrate, or sodium hydrate.—This produces a white amorphous precipitate which changes into crystals on standing or by rubbing the slide with a glass rod.

The crystals are rod-like, frequently with more or less chisel-like ends and a V-shaped recess extending backward into the crystal. There is a strong tendency to form coarse clusters up to about 15 branches. In open drops tree-like forms are frequent. For each of these reagents dilutions up to 1:1,000 give the reaction and the limit at 1:100 is 3 μ gr.

Sodium carbonate.—This gives a precipitate with cocain like that produced by potassium hydrate, both in the amorphous and crystalline forms. Limit of dilution is 1:1,000. In 1:100 solution the limit is 3 μ gr.

In order to determine the usefulness of some of the above tests when other alkaloids are present the palladous chlorid test was made on test drops to which had been added solutions of one of the following alkaloids, codein sulphate, atropin sulphate, heroin, dionin, acoin powder, cinchonin sulphate, hydroxylamin hydrochlorid, apomorphin hydrochlorate, narcotin, papaverin, brucin, narcein, morphin, thebain, gujasanol, orthoform (new), cinchonidia sulphate, quinidia sulphate, beta-eucain, holocain, caffen, quinin sulphate, strychnin, and tropacocain. In each case the crystals of the cocain compound were obtained and in the case of brucin, gujasanol, caffen, strychnin, and tropacocain, with which the palladous chlorid regularly gives a crystalline precipitate, it was found that when cocain was also present the cocain product was given in addition to that for the other alkaloid, though occasionally with modified form.

The foregoing serves to give an idea of the scope of the work undertaken, which it is hoped will be carried much further during the coming year.

COOPERATIVE WORK ON HEADACHE MIXTURES.

By W. O. EMERY.

After making investigations of various suggested methods for determining the different constituents present in the many headache mixtures containing acetanilid and similar agents, a method was finally devised which proved quite satisfactory to the members of the Division of Drugs, and it was therefore decided to place this method in the hands of as many chemists interested in this line of work as could assist. A circular letter requesting cooperation was sent out, and a gratifying number responded, signifying their willingness to assist, eleven of whom sent in results. All who expressed a desire to cooperate were supplied with a sample of a mixture containing known amounts of acetanilid, sodium bicarbonate, and caffen, with the following instructions, the U. S. Pharmacopœia, eighth revision, as amended and corrected May 1 and June 1, 1907, being used as a basis for all calculations and reagents unless otherwise specified:

SEPARATION OF CAFFEIN, ACETANILID, AND SODIUM BICARBONATE.

Caffen.

Weigh out about 0.3 gram of headache powder on a small (5.5 cm) tared filter,^a wash with successive small portions of chloroform to the amount of about 30 cc, collecting the solvent in a 100 cc Erlenmeyer. Distil off chloroform by means of a small flame until only a few cubic centimeters remain. Add 10 cc of dilute sulphuric acid, then continue the distillation till all the chloroform has gone over, disconnect from condenser, heat gently, first on wire gauze to complete solution,^b finally on a steam or hot-

^a In cases of powder mixtures or tablets containing ground celery seed, much coloring matter, cinchona alkaloids, laxative or extractive principles other than acetanilid or phenacetin, it is our practice to shake out the latter by means of chloroform from dilute sulphuric acid solution.

^b In case the preparation contains ground celery seed or certain oily principles, it sometimes happens that the acid solution does not become entirely clear at this point.

water bath until the contents of the flask have evaporated to about 3 to 4 cc. Cool, transfer by washing with water to a separatory funnel, so that the final volume does not greatly exceed 20 cc. Add four times the volume, or about 80 cc of chloroform, shake for some time vigorously, allow to stand until the chloroform clears perfectly, pass through a small dry filter into a dry 100 cc Erlenmeyer, distil off the solvent and use distillate for a second extraction, observing the same method of shaking, clearing, and filtering as above noted. Distil off chloroform to a small volume, transfer residue to a small tared beaker, or crystallizing dish, by means of a few cubic centimeters of chloroform. Allow to evaporate spontaneously, or if desired on a steam or hot-water bath to dryness, in the latter case partially covering the dish toward the end of operation with a watch glass in order to avoid possible loss from "popping." Cool in desiccator and weigh as caffeine, dry alkaloid.^a

Acetanilid.

First method.—The acid solution remaining in the separator and containing anilin sulphate is run into a 100 cc Erlenmeyer, the filter through which the chloroform passed is washed once with a little water, allowing the latter to run into the separator. Rinse the latter thoroughly, adding the aqueous rinsings to the acid solution. Now, run in slowly and with constant agitation a standard solution of potassium bromid-bromate ^b to a faint but distinct yellow coloration. The number of cubic centimeters employed, multiplied by the value of 1 cc in terms of acetanilid, will give the amount of acetanilid present.

Second method.—The acid solution aforesaid is treated with successive small portions of sodium bicarbonate until an excess of this reagent is observed in the bottom of the separator. Add 50 cc of chloroform and 15 to 20 drops of acetic anhydrid, shake for some time vigorously, allow the chloroform to clear, then pass through the same filter used for the caffeine into a 100 cc Erlenmeyer, and distil off most of the chloroform. Use this distillate for a second shake out, clear, filter, and distil down to a small volume, transferring the residue and the subsequent chloroform washings to a tared beaker or dish precisely as in the case of caffeine. Allow the solvent to evaporate spontaneously or by means of a blast or fan, avoiding, however, undue heat.^c Dry in desiccator over quicklime to constant weight.

Verify the final weight by means of titration with standard potassium bromid-bromate solution as in the first method. Heat the residue with 10 cc dilute sulphuric acid a half hour on the steam or vapor bath, cool, add 5 cc of water and titrate as directed above.

Sodium bicarbonate.

The residue left after the first treatment with chloroform is weighed when dry and represents very nearly the amount of sodium bicarbonate present. It may be more accurately estimated by titrating with tenth-normal sulphuric acid, using congo red as indicator, or it may be ignited with dilute sulphuric acid and weighed as sodium sulphate.

Calculate results in parts per 100.

^aShould the caffeine not be colorless or nearly so, the residue is dissolved in about 10 cc of water, filtered, if necessary (in case oily matters are present), through a wet filter, the filtrate acidified with dilute hydrochloric acid, the caffeine precipitated with 15 to 20 cc of Wagner's reagent, allowed to stand a half hour, filtered, and the precipitate washed with a few cubic centimeters of same reagent, the filter, together with precipitate, transferred to separator, decolorized by means of sodium sulphite, and the caffeine finally extracted with chloroform.

^bFor this purpose the solution is prepared by adding bromin in slight excess to a concentrated aqueous solution of 50 grams caustic potash, the liquid diluted till the separated salts redissolve, boiled, to expel any excess of bromin, and finally made up to 1 liter. This solution is standardized with weighed amounts of acetanilid, or it may be so adjusted by further dilution that 1 cc is exactly equivalent to 1 centigram of acetanilid. For purposes of titration 1 to 2 decigrams are heated a half hour on the steam or water bath with 10 cc of dilute sulphuric acid.

^cAcetanilid suffers appreciable loss when heated above 40°.

The results reported are tabulated as follows:

Results obtained in the cooperative work on an acetanilid mixture.

Analyst.	Caffein.	Acetanilid.		Soda bicarbonate.		Total. ^b
		Volumetric.	Gravimetric.	Volumetric.	Gravimetric.	
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
L. A. Brown, North Dakota.....	12.16	65.93		23.20		101.29
	10.93	66.10			23.50	100.53
	11.05	65.93			23.20	100.18
	10.40	66.10				
L. D. Havenhill, Kansas.....	11.50	63.00			25.10	99.60
	10.73	61.50			25.00	97.23
	11.33	63.40			24.90	99.63
	11.53	63.44			25.00	99.97
H. L. Schulz, Michigan.....	11.00	63.00			24.60	98.60
	11.00	62.00			24.80	97.80
H. A. Seil, New York.....	10.55	65.78			25.03	101.36
	10.49		65.26		25.11	100.86
	10.48	65.80			25.13	101.41
T. F. Darling, New York.....	10.62		67.72		25.13	103.47
E. L. Redfern, Nebraska.....	9.80	65.10			25.06	99.96
	10.06	64.58			24.93	99.57
	9.93	63.03	63.60		25.00	98.25
	10.20	63.21	63.80		24.93	98.64
E. M. Bailey, ^a Connecticut.....	10.90	64.53			25.07	100.50
	11.17	64.00			24.54	99.71
	11.30			25.93	25.57	
C. B. Morrison, ^a Connecticut.....	11.37			25.67	25.33	
	10.67	64.80		25.93	25.43	101.15
A. R. Mehrtens, California.....	10.30	64.53			24.99	99.82
G. E. Colby, California.....	10.00	65.33			24.79	100.12
W. O. Emery, Washington, D. C.....	10.23	64.79			24.92	99.94
	10.29	64.85			24.95	100.09
	10.01		64.68		25.01	99.70
Average.....	10.71	64.38	65.01	25.18	24.89	99.96
Maximum.....	12.16	66.10	67.72	25.93	25.57	103.47
Minimum.....	9.80	61.50	63.60	23.20	23.20	97.23
Difference.....	2.36	4.60	4.12	2.73	2.37	6.24
Known composition of acetanilid mixture (acetanilid, 453 parts; caffeine (anhyd.), 70 parts; soda bicarbonate, 174 parts)....	10.04	64.99		24.96		99.99

^a Reported by J. P. Street.

^b In cases where two percentages for volumetric and gravimetric determinations of the same substance were reported, the mean of such percentages has been taken in computing the total percentage.

Owing to an ambiguity in the expression "dilute sulphuric acid" employed in the method under caffeine, as also in the footnote *a*, page 100, for standard bromid-bromate solution, some of the workers quite naturally used the pharmacopœial strength, with the result that the acetanilid was not completely hydrolyzed. This undoubtedly explains the somewhat high results for caffeine and the correspondingly low ones for acetanilid. The strength of acid intended and the one actually employed for this purpose in the Bureau of Chemistry is that ordinarily used in laboratory work and is made by diluting 1 part of concentrated sulphuric acid (whose specific gravity is not less than 1.826 at 25°) with 5 parts of water. From two to three hours' heating on the steam bath is usually required to completely hydrolyze the acetanilid.

Notwithstanding this ambiguity the results obtained are very gratifying, in view of the fact that the method is new and the workers have entered into a comparatively new field. The percentages of variation are so small as to almost warrant the referee in recommending it as a provisional method to the association. He believes, however, that the method should receive additional study, and so recommends. It is also recommended that additional mixtures be tested with this and such other methods as may be found desirable.

President Snyder introduced the Secretary of Agriculture with a few words of appreciation concerning the long-sustained attitude of the Secretary in fostering agricultural chemistry, especially the work of the official chemists, by making possible the close affiliation between the Department of Agriculture and the association. The Secretary then briefly addressed the convention, after which the reading of the drug reports was resumed.

THE NECESSITY FOR ANIMAL EXPERIMENTATION IN DETERMINING THE PURITY AND STRENGTH OF MEDICINAL PREPARATIONS.

By WILLIAM SALANT.

Experiments on animals have long been recognized in medical jurisprudence as a valuable adjunct to chemical and microscopical methods in the detection of poisons in animal tissues and fluid. Notwithstanding the improvements in the methods of analytical chemistry witnessed within recent years, tests on animals, or, as Kobert ^a terms it, "biological testing," is still resorted to in order to corroborate the findings of the analytical chemist in cases of suspected poisoning with alkaloids and other poisonous substances of plant or animal origin. The French chemist, Boutmy, ^b who made extensive studies on poisoning with alkaloids, concluded that in all cases in which the presence of an alkaloid in the body is suspected experiments on animals should be made for the purpose of confirming the results of chemical analysis.

The work of some investigators indicates that the biological method is in certain cases much more delicate than the chemical. Ranke ^c reports experiments on dogs which were given 0.1 of a grain of strychnin by mouth. Chemical examination of the organs of these animals failed to show the presence of strychnin, but when extracts of the same organs were injected into frogs tetanus followed. Falck ^d has shown long ago that one-twentieth of a milligram of strychnin was sufficient to induce tetanus in a medium-sized frog. It might be added that if smaller frogs are used the same effect may be obtained with one-eightieth of a milligram. Atropin is another example of a drug of which small quantities are sufficient to produce a physiological reaction. Only one-twentieth of a milligram is necessary to produce dilation of the pupil.

Likewise cocain, which produces characteristic effects on the mucous membranes, and by its action on the frog's pupil, can be identified, even when very small quantities are present in biological solutions.

Aconitin can be identified in milligram doses by its action on the tongue, eye, heart, and central nervous system. No chemical methods have as yet been devised by which such small quantities of this alkaloid can be detected. A striking illustration of the delicacy of the biological method is afforded by the work of Hunt. ^e In his investigations on the functions of the thyroid he has shown that mice fed for a few days with the extract of this gland acquire greater resistance to poisoning with acetonitrile. One milligram of the official dried thyroid fed to white mice daily for a few days may enable the animals to recover from double the dose of acetonitrile fatal to the controls. Seidell, ^f working under the direction of Hunt, found that forty to fifty times as much thyroid would be required to give the iodine test.

^a Ber. deutsch. pharm. Ges., 1903, 13: 325.

^b Ann. hyg. publique med. legale, 1880, [3] 4: 193.

^c Virchow's Archiv, 1879, 75: 20.

^d Vierteljahrsschr. gericht. Med., N. F., 1874, 20: 198.

^e J. Amer. Med. Assoc., 1907, 49: 240.

^f Ibid.

Even more delicate are the methods employed in the study of immunity. As shown by the work of Meyer ^a and Uhlenhuth,^b by means of the precipitin test the presence and nature of proteins may be ascertained in a dilution of 1:100,000. As is well known, the chemical tests for albumin are of no value in dilutions over 1:1,000, and it is not specific.

For the identification of some poisons and the standardization of certain drugs of vast therapeutic application experiments on animals are practically the only reliable method.

There are no satisfactory chemical methods for the identification of the saponins, but owing to their powerful hemolytic action and their effect on the heart and voluntary muscles their identification has become possible. According to Kobert,^c picrotoxin can be identified by experiments on animals only. The detection of curarin by chemical tests is very unsatisfactory; by its action on the motor end organs, however, its identity can be established even when mere traces are present. Thus motor paralysis in frogs has been induced by injecting 0.005 milligram of curarin.

Adrenalin has been the subject of numerous investigations. On account of its powerful action and its extensive therapeutic application, the strength of the various preparations should be accurately known. A number of color tests have been proposed for this purpose, some of which are of doubtful value and some may be employed if properly controlled by tests on animals, which are very delicate. Meltzer and Auer ^d have shown that a drop of a solution of 1:120,000 dropped into the conjunctival sac of a rabbit causes blanching of the conjunctiva and dilation of the pupil. According to Ehrmann,^e a reaction of the pupil of the excised eye of the frog may be obtained with 0.000025 milligram of this drug. Similar results were obtained by other investigators who worked on the pharmacology of the drug. Cameron's ^f experiments with this drug on rabbits have shown that 0.0003 milligram per kilogram will cause a rise of blood pressure.

Preparations of digitalis have been found to vary enormously in physiological activity. Frankel ^g states that the strength of the tincture varies from 200 to 400 per cent, and the infusion varies from 100 to 125 per cent. In a recent article Lutzkaja ^h states that the infusion of digitalis he examined was only 60 per cent of the strength represented by the firm which prepared it. Barger and Shaw,ⁱ in England, examined nine tinctures of this drug, and found a variation of 75 per cent in their strength. These authors made a comparative study of Keller's method and the physiological test on an artificial infusion of digitalis made by adding a known quantity of the drug to a mixture of hay and chaff. This was extracted with 60 per cent alcohol. Chemical analysis of the extract showed the presence of 0.1 per cent of digitalis, whereas the mixture contained 0.4 per cent. The same extract was tested on frogs, however, and found to contain approximately 0.4 per cent.

In the case of some drugs the physiological test above must be relied upon, no chemical method having as yet been devised for their identification or quantitative determination. Cannabis belongs to this category. Houghton and Hamilton,^j in a recent article, state that previous to the adoption of this test, preparations of the

^a Lancet, 1900 (2), p. 98.

^b Deutsch. med. Wochenschr., 1900, 26: 734.

^c Loc. cit.

^d Centrbl. Physiol., 1904, 18: 317.

^e Arch. exp. Path. Pharmac., 1905, 53: 97.

^f Proc. Roy. Soc. Edinburgh, 1905, 26: 161.

^g Ther. Gegenwart, N. S., 1902, 4: 112.

^h Arch. intern. pharmacodynamic, 1908, 18: 77.

ⁱ Yearbook of Pharmacy, 1904, p. 541.

^j Ther. Gazette, 1908, 32: 26.

drug were so unreliable that hospital physicians used to experiment on patients with samples of this drug before placing an order for it. Since experiments on animals with this drug have been introduced, the practice of testing the preparations on human beings was abandoned.

Ergot is another drug whose activity can at present be determined only by experiments on animals. Crawford,^a who made a study of chemical tests, came to the conclusion that they are of no value, while its action on the cock's comb after subcutaneous injection, or on the isolated uterus of the cat, is characteristic and may therefore be used to advantage in its identification or in determination of the strength of the preparation.

REPORT ON INSECTICIDES.^b

By C. C. McDONNELL, *Referee*.

A study of methods for the examination of insecticides and fungicides was taken up by the association only ten years ago at the fifteenth annual convention, when the first referee on this subject was appointed.

For the two years following this no analytical work was reported, but methods were compiled and suggested for the examination of this class of materials which were then most important, and these were adopted provisionally. All of them have been tested since that time and those proving of value have been officially adopted by the association. A number however have since been replaced by more rapid and accurate methods. As the list of substances used as insecticides and fungicides increases, as it is constantly doing, new methods must be devised and changes in old methods introduced in order that they may be adapted to a particular material.

At the present time the most important of these is lead arsenate, and it is to methods for the examination of this substance that considerable study is now being given. The work as carried out this year has been largely along the line of the recommendations of the referee last year, which were adopted by the association. In addition a modification proposed by the present referee for the determination of total arsenic oxid in London purple has been given a trial.

WORK AS OUTLINED.

(1) A comparison of the provisional methods for London purple given in Bureau of Chemistry Circular 10, revised, the method as modified by Davidson, given in the Proceedings of the twenty-second annual convention of the association, also in Bureau of Chemistry Bulletin 107, revised, and the modified method as proposed by the present referee.

(2) A further study of the precipitation method for soda-lye, using fifth-normal acid instead of half-normal, also with and without removal of the barium carbonate precipitate before titration.

(3) Determination of formaldehyde in strong solution by the provisional hydrogen peroxid method, and on dilute solutions by the cyanid method to determine the amount of dilution necessary.

(4) Further test of the Avery method for the determination of sulphur in sulphur dips.

(5) A continuation of the study of the methods for lead arsenate proposed by Haywood (Proceedings 1906, Bulletin 105, p. 165) and tried last year.

Samples were sent to five chemists, who had expressed a willingness to cooperate in the work, and more or less complete reports have been received from three of these.

^a Amer. J. Pharm., 1908, 80: 326.

^b Owing to the illness of the referee, this report was not ready for presentation to the convention at the time of its meeting. Through the courtesy of the association the referee was permitted to present the report at a later date for insertion in the Proceedings.

LONDON PURPLE.

Owing to the considerable variation in results and the difficulties encountered in the carrying out of the present methods for the determination of total arsenic oxid in London purple, methods for this substance have been receiving considerable attention from the association for several years. The three principal objections to the methods thus far proposed are: (1) The difficulty in reading the end point when using up the excess of liberated iodine with sodium thiosulphate after the reduction of the arsenic oxid to arsenious oxid. (2) The difficulty in reading the end point in the final reaction on adding the standard iodine solution. (3) The great tendency to foaming on adding sodium carbonate to neutralize the acid. Both the first and second are caused entirely, and the third largely, by the great amount of organic matter (dye) contained in London purple. Several methods have been proposed, which are more or less successful, for overcoming these difficulties. The two which are most used, however, have not given satisfactory results in the hands of all the analysts using them, in many cases the results running several per cent too low. The modification proposed by the referee and tried this year overcomes these difficulties and renders the determination of total arsenic oxid much easier, particularly for one who has not had considerable experience with the present methods.

In the following tables are given the results obtained by the different analysts, followed by their comments:

TOTAL ARSENIUS OXID.

Method I is a provisional method and may be found in Bureau of Chemistry Bulletin 107, revised, page 28. Method II may be found on page 29 of the same bulletin. The results are very satisfactory, Method II appearing to give slightly lower figures.

Total arsenious oxid (As_2O_3).

Analyst.	Method I.	Method II.
	<i>Per cent.</i>	<i>Per cent.</i>
R. J. Davidson, Blacksburg, Va.....	22.10	21.90
	21.91	21.90
R. W. Thatcher, Pullman, Wash.....	22.45	21.99
	22.37	21.69
		22.01
		22.01
		22.38
C. D. Woods, Orono, Me.....		22.38
		21.74
		21.74
		21.74
	22.07	21.90
C. C. McDonnell, Bureau of Chemistry.....	22.23	21.83
	22.26	21.86
Average.....	22.20	21.93

TOTAL ARSENIC OXID.

Method I is a provisional method and may be found in Circular 10, revised, and also in Bulletin 107, revised, of the Bureau of Chemistry, page 28. Method II may be found in Circular 10, revised. Method III is Davidson's modification of Haywood's method for removing a part of the coloring matter and may be found in Bulletin 107, revised, where it is designated as Method II, Provisional (p. 29).

Method IV is that proposed by the referee and is as follows:

Place 2 grams of the sample in a 200 cc graduated flask, add 5 cc of concentrated nitric acid and 20 cc of concentrated sulphuric acid. Place on a hot plate or over low flame and heat nearly to boiling; after ten or fifteen minutes add powdered sodium nitrate, in small quantities at a time, until all organic matter is destroyed and the solution is colorless. Cool, add about 50 cc of water (to decompose any nitro-sul-

phuric acid formed), and heat to boiling till nitric acid fumes are all expelled. Cool, make up to mark with distilled water, mix thoroughly, filter through a dry filter and take 50 cc of the filtrate (0.5 gram) for the determination of arsenic oxid. Transfer this 50 cc portion to a 400 cc Erlenmeyer flask, dilute to about 100 cc with water, add 2 to 3 grams of potassium iodid and 5 cc of concentrated sulphuric acid, heat to boiling and evaporate to about 40 cc. Cool, dilute to 150 cc to 200 cc and add approximately tenth-normal sodium thiosulphate just to disappearance of color caused by the free iodine. In case the solution is slightly colored from iron or incomplete oxidation of the organic matter, add the thiosulphate until nearly colorless, then add a few drops of starch paste and continue adding the thiosulphate slowly until the blue color just disappears. The exact end point can easily be obtained in this way. Neutralize immediately with sodium carbonate, make slightly acid with dilute sulphuric acid, and when all lumps of sodium carbonate are dissolved add sodium bicarbonate in considerable excess. Titrate with twentieth-normal iodine solution in the usual way, using starch solution as indicator. Subtracting from this the number of cubic centimeters of iodine solution corresponding to arsenious oxid as determined by Method I, gives the number of cubic centimeters of iodine solution corresponding to the arsenic oxid (As_2O_3) in 0.5 gram of the sample.

Total arsenic oxid (As_2O_3).

Analyst.	Method I.	Method II.	Method III.	Method IV.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
R. J. Davidson, Blacksburg, Va.....	18.69	18.76	18.55	18.63
R. W. Thatcher, Pullman, Wash.....	17.53		18.55	19.01
	17.50		15.64	18.29
			15.56	18.06
			15.83	
			15.55	
			15.95	
C. D. Woods, Orono, Me.....			15.15	
			15.50	
			15.75	
			15.79	
			15.15	
C. C. McDonnell, Bureau of Chemistry.....	19.34	18.29	17.74	19.29
	19.73	18.23	18.04	19.42
	19.52	18.42	17.90	19.30

COMMENTS OF ANALYSTS AND DISCUSSION.

R. J. Davidson: There is no great difficulty in working the London purple by Method IV for arsenic oxid. It is perhaps a little more troublesome and you have to use another method for getting the arsenious oxid. I believe Method III, provided it gives satisfactory results, is the simplest method, both determinations being made from the same weighed sample.

R. W. Thatcher: Davidson's modification (Method III) makes the end point somewhat easier to determine. The modification suggested by the referee does not seem to me to offer any advantage over the official method, since the yellow color left after oxidation with nitrate mixture obscures the end reaction as badly as does the original color and has the disadvantage of requiring a separately weighed sample for the determination of the arsenic in arsenious form.

C. D. Woods: I would emphasize the fact and recommend that it be included in directions for insecticide work, that this kind of work is difficult for a beginner and that several preliminary determinations should be run before a man new to this work attempts to report results.

As has been the case in previous years, the results on arsenic oxid are very unsatisfactory, there being a difference of over 4 per cent between the highest and lowest determinations by Methods I and III, and over 2 per cent difference by the same method by different analysts. Methods II and III give lower results than Method I, but there does not appear to be any uniformity in the amount that these methods fall short, the determinations made by different analysts and even those by the same analyst at different times sometimes agreeing with those made by Method I and at others showing a variation of several per cent. Why this is so has not as yet been determined, but is under investigation. It is the referee's opinion that on precipi-

tating the coloring matter with sodium carbonate a varying amount of arsenic is carried down in the precipitate.

The determinations made by Method IV, while not agreeing as closely as might be desired, are close enough to justify a more extended trial of the method in the hands of different analysts. The writer has found it very satisfactory and, when properly carried out, a perfectly clear solution can almost always be obtained. Of course it is desirable to be able to determine both forms of arsenic on the same solution, but if it is found that this can not be done accurately this objection to the method becomes of minor importance.

LEAD ARSENATE.

The methods used for lead arsenate were proposed by Haywood at the meeting of the association in 1906 and were tried last year. They may be found in Bureau of Chemistry Bulletin 105, page 165; also Bulletin 107, revised, page 239.

The sample sent out for the work was made by the referee from C. P. di-sodium arsenate and lead acetate.

Lead arsenate.

Analyst.	Moisture.	Total arsenic oxid (As ₂ O ₅).	Total lead oxid (PbO).
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
R. J. Davidson, Blacksburg, Va.....	0.11	30.07	
	.12	30.07	
R. W. Thatcher, Pullman, Wash.....	.09	30.14	
	.09	29.97	
		30.39	68.38
		30.24	68.48
		29.63	66.31 65.96
		29.81	65.76 65.06
C. D. Woods, Orono, Me.....	.14	29.95	65.70 66.66
	.17	30.22	66.22 67.54
	.14	30.22	66.14 66.89
		30.22	65.94 67.17
		29.47	65.85 66.95
		29.80	66.58 67.32
C. C. McDonnell, Bureau of Chemistry.....	.15	29.83	67.55 67.38
	.17	29.60	67.35 67.00
			67.57 67.55
			67.58 67.10

^a Porcelain gooch used in all determinations of lead oxid.

DISCUSSION.

C. D. Woods states that in the determination of total arsenic oxid it was not found necessary to add thiosulphate to use up free iodine because if care is used in boiling the solution a colorless point is easily obtained.

The results on lead arsenate are not so uniform as might be desired, particularly on total lead oxid. However, the difference between the highest and lowest determination of arsenic oxid is only 3 per cent of the total amount present and for lead oxid 5 per cent of the total amount present. The method is certainly the best that has thus far been proposed and if carefully followed good results should be obtained.

SODA LYE.

METHOD I.—This is the precipitation method, and may be found in Circular 10, revised, page 8, and Bulletin 107, revised, page 31.

METHOD II.—This is the same as Method I except that the titration for hydroxide is made without removing the barium carbonate precipitate.

The acid potassium sulphate method was not submitted for trial, as satisfactory results had not been obtained by it in previous years and the association voted that it be dropped, as recommended by the referee in 1907.

As it was desired to send out samples containing considerable carbonate, and such were not at hand they were prepared as follows: The sample bottle was weighed and into this was weighed 2 grams dry sodium carbonate C. P. then, as rapidly as possible, 18 grams of commercial sodium hydrate. The bottles were then stoppered and sealed.

The analyst was directed to dissolve the entire content of the bottle in carbon dioxide-free water, make up to 2,000 cc and use 50 cc portions for the titrations (0.5 gram sample). The results submitted have been multiplied by two and reported in per cent in the following table:

Soda lye.

Analyst.	Method I.		Method II.	
	Sodium hydroxid (NaOH).	Sodium carbonate (Na ₂ CO ₃).	Sodium hydroxid (NaOH).	Sodium carbonate (Na ₂ CO ₃).
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
R. J. Davidson, Blacksburg, Va.....	84.40	13.25	84.80	12.72
R. W. Thatcher, Pullman, Wash.....	83.36	14.92	83.68	14.50
	83.76	14.40	83.52	14.74
C. C. McDonnell, Bureau of Chemistry.....	84.72	11.80	84.92	11.54
	84.72	11.80	84.92	11.54
Average.....	84.19	13.23	84.37	13.07

The results on sodium hydroxid are very good. As expected, Method II gives slightly higher results for hydroxid and lower on carbonate than Method I. The difference, however, is small. The referee determined carbon dioxide in a portion of the sample gravimetrically and found 11.62 per cent and 11.71 per cent calculated as sodium carbonate.

Using these two indicators in the same determination, as is done in this method, the tendency would always be to high results on sodium carbonate. Phenolphthalein, being more sensitive to acids, becomes colorless immediately when the solution is neutral, while with methyl-orange the acid must be in slight excess to develop the pink color, the excess required depending on the amount of indicator used and the depth of color titrated to. A blank should be made, using the same amount of water and indicator, and deducted in each case when methyl-orange is used. For the determinations in the second report in the table the analyst used normal acid. This may account for the results in sodium carbonate being high, as 0.1 cc normal acid is equivalent to over 1 per cent sodium carbonate, when operating on 0.5 gram of substance.

FORMALDEHYDE.

Two samples were sent out for analysis, No. 1, a strong solution to be worked by the modified hydrogen peroxid method, and No. 2, a dilute solution to be worked by the cyanid method, both found in Bulletin 107, revised, page 33.

Formaldehyde.

Analyst.	Sample No. 1. Method I.	Sample No. 2. Method II.
	<i>Per cent.</i>	<i>Per cent.</i>
R. J. Davidson, Blacksburg, Va.....	36.81	3.92
	36.71	3.98
R. W. Thatcher, Pullman, Wash.....	36.64	3.92
	36.46	3.83
		3.70
C. C. McDonnell, Bureau of Chemistry.....	37.00	3.98
	36.93	3.84
		4.02
Average.....	36.76	3.90

COMMENTS AND DISCUSSION.

R. J. Davidson says: "I believe it would be well to state the amount of dilution necessary in Method II and not say, as the method does, 'a weighed quantity of the dilute formaldehyde solution.' The directions should be more specific."

The results on formaldehyde are very good. Method I is an excellent method for strong solutions, and Method II for dilute solutions, containing preferably not over 5 per cent. Even solutions of the latter strength must be diluted before making the determinations.

The referee is in favor of the recommendation made last year and referred to again in Mr. Davidson's report, that more specific directions should be given this method. If, instead of the words "a weighed quantity of the dilute formaldehyde solution," line 8, the following were inserted, "a weighed quantity of the formaldehyde solution containing not over 2 cc of a 1 per cent solution or the equivalent," it would make the method clearer and sufficiently explicit.

SULPHUR DIPS.

The method is that of Avery and is given in Circular 10, revised, also Bulletin 107, revised, page 34. The sample submitted for analysis was prepared in the laboratory by boiling together lime and sulphur according to the regular formula for the lime-sulphur spray mixture.

Sulphur dips.

Analyst.	Date of analysis.	Weight of sulphur.
	1908.	<i>Gram per cc.</i>
R. J. Davidson, Blacksburg, Va.....	August 18.....	0.03452
		.03455
		.03452
R. W. Thatcher, Pullman, Wash.....	July 9.....	.03683
		.03684
		.03696
C. C. McDonnell, Bureau of Chemistry.....	July 20.....	.03536
		.03583
		.03575
Average.....		.03568

The results are all very close, the greatest difference being only 0.25 per cent. This method has also given satisfactory results in past years.

In view of the fact that this report was not presented at the meeting of the association, no recommendations will be made at this time.

PRESIDENT SNYDER'S ADDRESS: THE TRAINING OF THE AGRICULTURAL CHEMIST.

I have selected as the subject of the president's address for this, the twenty-fifth annual convention of the Association of Official Agricultural Chemists, "The Training of the Agricultural Chemist."

Any society or organization in order to be effectual and progressive must look well to its membership. Our society has been most fortunate in this respect, and it is to be hoped its ranks will continue to be filled with the same class of earnest, energetic workers as are here to-day. During the past quarter of a century this organization has accomplished most excellent results. I believe, however, that it has only entered upon its career of usefulness. Much credit is due to the founders for the high ideals of the association and for the cultivation of the true scientific spirit. Many of them

received their training in the great European laboratories, where they were students of Liebig, Fresenius, Voit, Hoffman, and Pasteur, and they have planted in this country the seed of true agricultural research. Most of the older members have relinquished their labors, and the work of the society may now be said to be in the hands of the second generation, who, it is hoped, will meet with as much success and foster the same spirit and ideals.

Originally agricultural chemists were in a way self-educated. They secured what knowledge they could of general and analytical chemistry and then applied it to the solution of agricultural problems. Naturally the work was largely analytical. "What does this substance contain?" was and is to-day the quest of the chemist. During the past few years, however, the domains of agricultural chemistry have been greatly enlarged and there is probably now no other branch of chemistry that calls for so wide a training. Organic, inorganic, industrial, physical, physiological, and sanitary chemists have definite channels within which their activities are confined, while the agricultural chemist must necessarily include in his domain a large portion of all of these. In dealing with the soil an extended knowledge of both inorganic and organic chemistry as well as of physical chemistry is requisite. Our knowledge of soils is necessarily much restricted because the chemistry of the silicates is so imperfectly understood, and so in the analysis of plant and animal substances and the interpretation of the results our knowledge is likewise very limited. While the data gained from the analysis of foodstuffs is exceedingly valuable, I do not believe that it is as much so as it is destined to be, and while chemistry is one of the most useful of the sciences in the study of agricultural problems, it is capable of being made still more valuable and useful.

One of the chief functions of the agricultural chemist is to correctly analyze agricultural products. In order to do this methods of analysis based upon rational principles must be devised, and this is one of the principal features of the work of this association. It is scarcely necessary for me to dwell upon its importance. Correct methods of analysis are essential, as without these chemistry would be entitled to no higher rank than alchemy. I do not believe that the importance of the development of correct methods for the analysis of agricultural products is as fully appreciated by experiment station workers as it should be. A large amount of the work that has been done is destined to be discredited and discarded because of errors in methods employed. Some of our experiment stations have been too impatient to secure immediate results and have not paid sufficient attention to methods of investigation. The study of the methods for analysis of foods and agricultural products can well be continued as the most prominent feature of this organization.

With the advance that is being made in general science and the greater recognition given agriculture, more extended provision should be made for the education and training of the prospective agricultural chemist. There are many institutions that offer excellent four-year courses in chemical engineering and other branches of chemistry leading to degrees. I know of no American institution where such a course is given in agricultural chemistry. Has not the time arrived for the establishment, in some of our institutions of courses of study having for their object the training of agricultural chemists? Certainly the importance and magnitude of the field would suggest the need of such courses. I shall not discuss the subjects that could most consistently form a part of the curriculum, but there should be a correlation of the different sciences blended with general and technical chemistry. As matters now stand, it is generally necessary for an experiment station to secure as assistants young chemists who have had but little training in analytical chemistry and give them special training in agricultural analysis. The experiment stations have to train their own assistants and by the time they have become reasonably proficient another institution or some industry offers a higher salary and then new assistants must be initiated, the process in some cases being repeated several times a year. Our

research work suffers because of this condition. Experiments are undertaken with one corps of assistants, a part of the work is done by another, and if the investigation is completed at all it is after many changes have been made. If some of our larger institutions would furnish more extended training in agricultural chemistry and better remuneration were given assistants so as to retain their services, conditions would be greatly improved. I do not consider that this lack of training of assistants is necessarily the fault of agricultural colleges, as their courses of study have been formulated with other objects in view than the training of scientists for research work. There are many interesting problems in agricultural chemistry which await investigation, and their correct solution would be of great benefit to mankind. The field of research is so large that this association can consistently encourage a larger number of workers.

In addition to the special technical training the agricultural chemist needs broad equipment in other lines so that he may be able to inaugurate useful lines of research and properly interpret his results. There are many chemists who are capable of making accurate and rapid analyses and prosecuting routine work, but are unable to outline an investigation, plan intricate details, carry the work to a satisfactory conclusion, and correctly interpret the results. There need be no fear of overcrowding in the realm of agricultural chemistry or necessity for forming a trade union to regulate the number practicing the profession. In this connection it is pleasing to note the greater recognition that is being given the agricultural chemist. About a decade ago the number of positions in this line were limited and the compensation exceedingly small. While neither the number of positions nor the compensation is now particularly large there has certainly been a material increase in both. For example, in the Department of Agriculture in 1897 the maximum salary paid was \$2,500 per year and the average to 12 chemists was \$1,541, while in 1907 the maximum salary was considerably greater and 47 chemists received an average of nearly \$2,000. On the whole, however, these salaries are smaller than are paid in many of the large educational institutions, although the rate of increase during the past ten years has been greater than in educational institutions, and if this continues the agricultural chemist bids fair in the near future to receive as large a compensation as workers in other lines of science. Much credit is due to our present Secretary of Agriculture for recognizing the importance of agricultural research and having the courage to advocate and recommend to Congress suitable compensation for agricultural scientists.

The position of the agricultural chemist in both the educational and business world is undergoing transition. He is being regarded as a greater factor in human and industrial progress than heretofore and I believe that with each decade he may reasonably expect greater opportunity to do good work, coupled with better compensation. Agricultural chemists have as a rule been underpaid; neither have they been given sufficient funds with which to prosecute their labors. In many laboratories bookshelves are not filled as they should be and makeshift apparatus is employed where better results could be secured if the chemist had at his command the literature covering the work of others upon the subject which he is investigating, and suitable apparatus and means for his work. There has been many a scientific surrender because of lack of funds for effectually carrying on the work.

As a nation we have taken great pride in the progress made by our industries, an advance more rapid than that of any other country. This in a large measure has been due to the work of the American chemist. There is scarcely an important industry but employs a well-trained chemist and has a suitably equipped testing laboratory. The steel, sugar, cement, and other great industries are practically applied chemistry. It has been said that the American chemist has contributed less than his quota to the advancement of science; he has, however, contributed his full share to the advancement of our industries. Instead of being a devotee of pure science he has advanced the domains of applied science. The agricultural chemist should

concern himself not only with the economic production of foodstuffs but should extend his work along the lines of their preparation and utilization. The production of food, while a very large and important subject, has associated with it its proper manufacture and utilization. The agricultural chemist should take a broader view than that of mere critic of the industries; and there is some danger when working along special food lines of his becoming too narrow in his consideration of the questions that present themselves. While adulteration and sophistication in any form should not be tolerated by the chemist, he should first make sure that it is adulteration, and in this connection there are destined to arise questions upon which scientific men materially and honestly differ. I should not care to see all scientists agree on all questions, as this would be detrimental to progress. There must be some attrition, and when differences arise they should be met in the true scientific spirit, each one being sure that the data and facts which he presents are absolutely correct in every detail. I believe the province of the chemist is first doing accurate analytical work. The stand which has been taken by this association is a most excellent one: That the meetings shall be open for discussion, that we invite thorough discussion of all subjects relating to the analysis of our agricultural products and the interpretation of their results, but that the views expressed by any one individual are not necessarily the official views of the association. In controversial questions it is well for the society to be conservative. We all recall the attempt of the French Academy of Science to settle the much-vexed question of atmospheric nitrogen as a source of plant food, and how, after examining the conflicting reports of Ville and Boussingault, the learned committee of the society reported that M. Ville's conclusions and results were consistent with his experiments. We well know how the conflicting work of these two investigators was later harmonized, and while the society attempted to decide the question the real question was not settled until years later. The best service this society can render the cause of agriculture is to continue along the lines followed by the founders, to improve the methods of analysis so that the work done by the official methods of the Association of Official Agricultural Chemists will command respect wherever quoted.

The food chemist should make a more careful and extended study of processes employed in the manufacture of foods. A purely theoretical knowledge of manufacturing processes may lead to erroneous conclusions. Some manufacturers of foods are doing more in the way of scientific investigation than are many of our universities and experiment stations. The encouragement given by the industries for the investigation of scientific subjects has been productive of fruitful results. Pasteur's classical work on fermentation was made possible by his connection with the industries. The agricultural chemist needs the help and assistance of the technical chemist.

One of our great needs is more funds with which to prosecute scientific inquiry. Men of science have the zeal and ability, but often fail for lack of funds to procure and construct scientific apparatus. And too often men in our universities who are specially adapted by nature for the prosecution of scientific investigations are overburdened with elementary instruction that could be more efficiently done by others. Many scientists attempt to do too much, and the result is a dissipation of energy.

Scientific work often suffers, too, because of the natural modesty of scientists, and sometimes those who accomplish the least but make the most noise, secure the lion's share of the funds for carrying on work. Some pseudo-scientists resort to cheap advertising that can not be too severely condemned. The best advertising a scientist can do is the publication of high-grade scientific work. It is a slow but a sure way of building up a permanent reputation. A scientist who maintains a press agency is not destined to make a premanent record.

Often science languishes because those immediately in authority are not sufficiently educated or naturally liberal enough to appreciate her claims or able to give wise and

intelligent direction to scientific investigations. Science should be completely segregated from politics as it is sometimes practiced, and she should not be dependent for her existence upon the whims of the spoilsman.

Science seeks to determine the truth. True science will not tolerate a falsehood nor perpetrate a fraud, and there is no place for the drone in the ranks of science; there have been a few who have made some progress by conjuring with scientific terms, looking wise, cultivating society, and catering to the whims of those in temporary authority and neglecting science. Others have had a brief but precarious existence as scientific pirates, appropriating to themselves the work and results of others, sometimes of advanced students and underpaid and dependent assistants. All true teachers and investigators enjoy having their assistants and students do good work and secure noteworthy results. A true scientist can honestly rejoice at seeing his colleague or coworker make a discovery. Petty jealousies are unworthy of science.

Agricultural chemistry is a great constructive agency and wealth producer. We are building our science for review by future generations. Let us build it well so as not to be ashamed of the workmanship. The true scientist bequeaths to mankind an invaluable legacy. Let us cultivate true science and not false ideals.

The proposed changes in the constitution, which had been made special order following the president's address, were considered. These changes were as follows:

In article 1, first sentence, substitute for the words "the United States," the words "North America."

In article 2, first sentence, second and third lines, insert the word "provincial" after the word "State;" also, in the third line of this sentence insert after the word "body" the phrase "in North America."

After discussion, the amendments were put to a vote and were carried.

A motion was made by Mr. Wiley to the effect that a referee and an associate referee on water analysis be appointed to study mineral, sanitary, irrigation, and technical waters, inasmuch as, under the food and drugs act, standard methods for potable waters were needed as well as for foods, while the analysis of irrigation waters was a purely agricultural question and also needed study and elaboration.

The motion that such referees be appointed was carried.

FRIDAY—AFTERNOON SESSION.

REPORT ON SOILS.

By S. D. AVERITT, *Referee*.

The association at its last meeting made only two recommendations affecting the referee's work this year.

- (1) That the modified J. L. Smith method for total potassium be further tested.
- (2) That the sodium peroxid fusion method for total phosphorus be adopted as a provisional method of this association and be further tested.

Including this year, these methods have been before the association three years, and it seemed very desirable to place before the association work sufficient in quantity and of such a quality as to enable it to dispose of them. From his experience as

a cooperator in the past, the referee thought it best not to ask for too much work, and in accordance with this view only one other line of investigation was requested of those who expressed a willingness to cooperate. In the opinion of the referee, the sodium peroxid fusion method for total phosphorus had some very serious disadvantages, particularly as to manipulation and length of time required for the determination. It was thought desirable to ask that a method in use in this laboratory be tested with a view to proposing it as a provisional method for total phosphorus in soils.

This method, which may be known as the magnesium nitrate method, is easy of manipulation and rapid, and in this laboratory has given uniformly as good results as the sodium peroxid fusion. Accordingly, it was asked that these two methods be compared.

Two well-known Kentucky soils were selected for this work. No. 1 is a cultivated soil from the western coal field. A complete analysis made by this station shows it to be poor in phosphates, organic matter, and nitrogen. No. 2 is a virgin soil from the Devonian in the eastern part of Clark County, known by analysis, as in the case of No. 1, to be particularly rich in phosphates, organic matter, and potash.

Samples were prepared and sent to fourteen chemists, who volunteered to aid in this work, with the following instructions:

INSTRUCTIONS.

(a) Make a determination of moisture by the official method, reporting the percentage.

(b) Weigh 10 grams of sodium peroxid into an iron or porcelain crucible and thoroughly mix with it 5 grams of the soil. If the soil is very low in organic matter, add a little starch to hasten the action. Heat the mixture carefully by applying the flame of a Bunsen burner directly upon the surface of the charge and the sides of the crucible until the action starts. Cover crucible until the reaction is over, and keep at a low red heat for fifteen minutes; do not allow fusion to take place. By means of a large funnel and a stream of hot water transfer the charge to a 500 cc measuring flask. Acidify with hydrochloric acid and boil. Let cool and make up to the mark. If the action has taken place properly there should be no particles of undecomposed soil in the bottom of the flask. Allow the silica to settle and draw off 200 cc of the clear solution.

Precipitate the iron, alumina, and phosphorus with ammonium hydroxid; filter, wash, return the precipitate to the beaker with a stream of water, holding the funnel over the beaker, and dissolve the precipitate in hot hydrochloric acid, pouring the acid upon the filter to dissolve any precipitate remaining. Evaporate the solution and washings to complete dryness on the water bath. Take up with dilute hydrochloric acid, heating if necessary, and filter out the silica. Evaporate filtrate and washings to about 10 cc, add 2 cc of concentrated nitric acid, and just neutralize with ammonium hydroxid. Clear up with nitric acid, avoiding an excess. Heat from 40° to 50° on water bath, add 15 cc of molybdic solution, keeping at this temperature for from one to two hours. Let stand overnight, filter, and wash free of acid with 0.1 per cent solution of ammonium nitrate; finally, once or twice with cold water. Transfer filter to beaker and dissolve in standard potassium hydroxid (1 cc equal to 0.2 mg of phosphorus), titrate the excess of potassium hydroxid with standard nitric acid, using phenolphthalein as indicator.

(c) Weigh into a 50 cc porcelain dish 5 grams of soil. Moisten with 5 to 7 cc of magnesium nitrate solution (*(g)* p. 2, Bul. 107, Bureau of Chemistry). Bring to dryness on water bath, burn off the organic matter at low redness; when cool, moisten slightly with water, add 10 cc of concentrated hydrochloric acid, digest two hours on water bath, keeping the dish covered with a watch glass; stir up two or three times during digestion.

Make up to 250 cc, mix well and throw on a dry folded filter, pouring back on the filter till the solution runs through clear. Take aliquots corresponding to 2 or 4 grams (4 grams in No. 1, 2 grams in No. 2), depending upon the amount of phosphorus present. Bring to dryness, take up with hydrochloric acid and water, filtering over pump. Filtrate and washings should not exceed 30 or 40 cc. Make alkaline with ammonia, and dissolve the precipitate with concentrated nitric acid, using a slight excess. Add gradually, while shaking, 5 to 15 cc molybdate solution (p. 2, Bul. 107). After standing a minute or two add 15 cc of ammonium nitrate (p. 2,

Bul. 107), shaking thoroughly. The solution should be kept at 40° or 50° C. for an hour, then let stand overnight at the room temperature, filter, and wash well with cold water. A Hirsch funnel with a double qualitative filter, S and S No. 597, cut to fit, and well pressed down around the edge with the finger after wetting and putting on pressure, is recommended. Put filter and precipitate back into the same flask, using as little water as possible for washing back into flask. Determine phosphorus volumetrically, using standard potassium hydroxid and nitric acid.

(d) Fuse 1 gram of soil according to the well known J. Lawrence Smith method. Transfer the fused mass to a porcelain dish, slake with hot water, grind finely with an agate pestle, and transfer to a filter. After washing free of chlorids, concentrate the filtrate and washings in a Jena beaker to about 20 cc, and filter. Slightly acidify the filtrate and washings with hydrochloric acid, concentrate in a platinum dish, add 1.5 cc of a platinic chlorid solution (10 cc contains 1 gram of platinum). Evaporate to a sirupy consistency, as usual, and wash with 80 per cent alcohol and ammonium chlorid solution.

(e) Determine potassium according to the regular J. Lawrence Smith method.^a

The referee inclosed with his instructions to those who expressed a willingness to cooperate in the work a short personal letter, and had hoped that each one would contribute something to this report, but unfortunately, as it often happens, many were not able to send in results in time for use. The referee desires to express his thanks to the following chemists who have aided him in the work: A. W. Gregory, for the Illinois station; W. P. Kelly, for the Hawaii station; P. E. Brown, for the New Jersey station; G. S. Fraps, for the Texas station; W. B. Ellett, for the Virginia station; I. O. Schaub, for the Iowa station; and P. F. Trowbridge, for the Missouri station.

It will be seen from Table 1 that the results obtained by the sodium peroxid fusion and the magnesium nitrate method show practically no difference. One chemist gets results in Soil II somewhat higher than the others, but the amounts of phosphorus obtained by the two methods on three determinations are almost identical. In Soil I, with the exception of one determination, the maximum and minimum results are not bad duplicates.

^a Fresenius's Quantitative Chemical Analysis, p. 426.

TABLE 1.—*Comparison of sodium peroxid fusion and magnesium nitrate methods for total phosphorus.*

[Water-free basis.]

Analyst.	Soil No. I.		Soil No. II.	
	Sodium peroxid.	Magnesium nitrate.	Sodium peroxid.	Magnesium nitrate.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
A. W. Gregory, Illinois.....	0.029 .028	0.027 .027	0.211 .210	0.224 .226
Average.....	.029	.027	.211	.225
W. P. Kelley, Hawaii.....		.030 .030 .031		.240 .244 .236
Average.....		.030		.240
P. E. Brown, New Jersey.....	.021 .029 .025 .025	.025 .025 .029	.281 .281 .285	.281 .285 .285
Average.....	.025	.026	.282	.284
E. C. Carlyle, Texas.....		.025 .023		.197 .201
Average.....		.024		.199
I. O. Schaub, Iowa ^a		.025		.213
W. B. Ellett, Virginia.....	.025 .024	.027 .028	.231 .230	.232 .232
Average.....	.025	.027	.231	.232
S. D. Averitt, Kentucky.....	.031 .032	.030 .030	.220 .213	.227 .229
Average.....	.031	.030	.217	.228
P. F. Trowbridge, Missouri ^a	.030	.029	.242	.219
General average.....	.028	.028	.237	.230

^a Duplicates not reported.

The referee did not ask that these methods be checked by the carbonate fusion or standard method, as it is sometimes called, his work with the method having shown that it has the disadvantage of a large amount of soluble silica, necessitating dehydration, and in the case of a soil with any considerable amount of organic matter there exist favorable conditions for reduction and, consequently, some loss of phosphorus.

In Table 2 will be found the results of the referee's determination of total phosphorus in the two soils, working as follows: Making a carbonate fusion, then proceeding as for an accurate determination of silica, the silica evaporated with hydrofluoric acid, the residue taken up with hot, strong hydrochloric acid and added to the filtrate from the silica. Iron, alumina, and phosphorus precipitated with ammonium hydroxid, washed, redissolved, and the phosphorus determined volumetrically, as in the magnesium nitrate method.

It will be seen from the table that in Soil I, containing very little organic matter, the results compare very favorably with the other methods, but in Soil II, rich in organic matter, the average is lower.

TABLE 2.—*Duplicate determinations of phosphorus by carbonate fusion.*

[Water-free basis.]

Soil I.	Soil II.
<i>Per cent.</i>	<i>Per cent.</i>
0.025	0.022
.033	.220
.026	.207
.....	.207
.028	.209

In Table 3 will be found duplicate determinations of total phosphorus in soils by the magnesium nitrate digestion. Of these, Nos. 1127, 1202, and 1204 are presumably Texas soils, and the determinations were made by Mr. E. C. Carlyle, of the Texas station. The others are low phosphate soils of western Kentucky and were made as checks in the general work of this laboratory.

TABLE 3.—*Duplicate determinations of phosphorus by magnesium nitrate method.*

[Water-free basis.]

Soil No.	Determination 1.	Determination 2.	Soil No.	Determination 1.	Determination 2.
	<i>Per cent.</i>	<i>Per cent.</i>		<i>Per cent.</i>	<i>Per cent.</i>
13	0.030	0.027	120	0.034	0.034
14	.051	.052	600	.035	.035
15	.046	.040	601	.043	.039
16	.052	.053	602	.028	.026
117	.046	.045	1127	.029	.027
118	.045	.045	1202	.031	.032
119	.034	.034	1204	.020	.021

Table 4 shows the modified method to compare very favorably with the regular J. Lawrence Smith method for total potassium, the former method giving in the general average 0.01 per cent more potassium in both soils. Taking into consideration the fact that the work is done on 1 gram the agreement is as close as could be expected. Referring to the work done by these methods in 1906 and 1907, it will be seen that the results are in the main concordant.

TABLE 4.—Comparison of modified and Smith methods for total potassium.

[Water-free basis.]

Analyst.	Soil I.		Soil II.	
	Modified.	Smith.	Modified.	Smith.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
A. W. Gregory, Illinois.....	1.156	1.205	1.568	1.582
	1.157	1.175	1.547	1.568
	1.182	1.164	1.566	1.594
	1.156	1.208	1.552	1.586
	1.158	1.223	1.547	1.566
Average.....	1.162	1.195	1.556	1.585
W. B. Ellett, Virginia.....	1.068	1.183	1.495	1.564
	1.081	1.121	1.487	1.536
Average.....	1.075	1.152	1.491	1.550
S. D. Averitt, Kentucky.....	1.190	1.162	1.552	1.491
	1.185	1.170	1.546	1.541
	1.214	1.202	1.531	1.565
Average.....	1.196	1.178	1.543	1.532
P. F. Trowbridge, Missouri ^a	1.177	1.186	1.512	^c 1.357
A. A. Wells, Iowa.....	1.271	1.121	1.608	1.751
	1.275	1.134	1.702	1.760
Average.....	1.273	1.127	1.655	^c 1.755
O. M. Shedd, Kentucky ^b				1.511
				1.627
Average.....				1.569
I. O. Schaub, Iowa.....	1.320		1.656	
	1.440		1.678	
Average.....	^c 1.380		1.667	
General average.....	1.177	1.168	1.571	1.556

^a Duplicates not reported.^b Too late to be included in the general average.^c Not included in the general average.

COMMENTS OF ANALYSTS.

P. E. Brown: The magnesium nitrate method is undoubtedly quicker and easier of manipulation than the peroxid fusion method. It has the advantage that there is not nearly such a large amount of silica to get rid of, as it was found necessary to dehydrate three or four times with the peroxid fusion method. Then, too, in the first operation of the fusion method there seems to be an uncertainty of reaction while avoiding fusion, which is of course eliminated in the other method.

You will notice from the results that the agreement is fair with a tendency for the new method to give slightly higher results. However, if the first determination in Soil I by the fusion method is eliminated, the agreement is much better. On the whole the magnesium nitrate method seems to me to be undoubtedly superior to the other.

W. P. Kelley: I find the magnesium nitrate method as outlined by you to be a very simple and convenient scheme for determining the phosphoric acid in soils; and while I have not had an opportunity to compare this method with others, I have no doubt that the results are reliable.

RECOMMENDATIONS.

The work done this year, while not as extensive as the referee had wished, still warrants in his opinion three conclusions, especially when it is remembered that work along the same line last year and the year before is mainly concordant in the matter of results: First, that the modified J. Lawrence Smith method for total potassium compares very favorably with the regular method and is somewhat shorter; second,

that the sodium peroxid fusion method for total phosphorus gives good results, but the manipulation presents some difficulty, and the time required for making the determinations is a disadvantage; third, that the magnesium nitrate method gives uniformly as good results for total phosphorus as the sodium peroxid fusion, and is quick and easy of manipulation. With these facts in view, the referee would make the following recommendations:

(1) That the modified J. L. Smith method for total potassium be adopted as an optional method of this association.

(2) That the sodium peroxid fusion for total phosphorus be adopted as an official method.

(3) That the magnesium nitrate method for total phosphorus be adopted as a provisional method of this association and be further tested.

REPORT ON THE DETERMINATION OF CALCIUM CARBONATE IN SOILS.

By JACOB G. LIPMAN, *Associate Referee.*

Systematic determinations of calcium carbonate in cultivated soils seem highly desirable in view of its important functions in crop production. Unfortunately, there is no unanimity of opinion among chemists as to the methods best adapted for this work. When the proportion of calcium and magnesium carbonates exceeds 1 per cent, fairly accurate determinations may be made by the liberation of carbon dioxide and its absorption and weighing in potash solutions. But when the proportion of carbonate is small, as is true of so many of our soils, the quantity of carbon dioxide which remains in solution in the acid is very large in proportion to its entire amount. This source of error has frequently been commented upon and has led to several more or less successful attempts to correct it.^a

The associate referee on soils thought it advisable, therefore, to outline some cooperative work on one or two promising methods for the determination of carbonates in soils. Samples of two different soils were sent to eleven members of the association who had signified their willingness to cooperate in the testing of soil-analytical methods. It was suggested that determinations of carbonates be made in the samples by Knorr's method as described in Wiley's *Agricultural Analysis*.^b Where possible, the results secured by Knorr's method were to be checked by the method described by Amos in the *Journal of Agricultural Science*.^c

The samples were sent out early in September, and analyses were made and reported by W. B. Ellett of the Virginia station, by Percy E. Brown of the New Jersey station, and Ernest Van Alstine of the Illinois station. Mr. Van Alstine's data were transmitted to the associate referee by Mr. Hopkins.

Mr. Ellett and Mr. Brown used Knorr's apparatus for the determination of the carbon dioxide. Mr. Van Alstine employed the method regularly used at the Illinois station and consisting of the liberation of the carbon dioxide "by boiling with hydrochloric acid and ascertaining the quantity of carbon dioxide evolved by measuring before and after absorption by a caustic potash solution." The results were as follows:

^a See Hall and Russel, *A Method for Determining Small Quantities of Carbonates*, Transactions, J. Chem. Soc., London, 1902, 81:81.

^b Vol. 1, p. 338.

^c 1905, 1:322.

Determination of carbon dioxide in soils.

[Percentage of dry soil.]

Analyst.	Soil No. 1.	Soil No. 2.
F. Van Alstine, Illinois.....	0.025 .025 .027 .024	0.020 .020 .020 .020
Average.....	.025	.020
W. B. Ellett, Virginia.....	.034 .030	.022 .025
Average.....	.032	.023
P. E. Brown, New Jersey.....	.031 .034 .030	.022 .026 .021
Average.....	.032	.023

The results submitted by Ellett and Brown agree very satisfactorily. Those submitted by Van Alstine are markedly lower, especially in the case of soil No. 2. Apparently the amount of carbon dioxide which remained in solution in the latter work is the cause of the lower results. Evidently Knorr's apparatus is efficient for the determination of comparatively slight amounts of carbonates; however, it is desirable that further work be done along this line, and the associate referee would therefore recommend that it be continued with certain modifications for at least another year.

REPORT ON POTASH.By B. B. Ross, *Referee*.

The work on potash for the past year has included cooperative tests of the regular official method in comparison with the phosphomolybdic volumetric method, and, in addition, the referee, associate referee, and some cooperating chemists have made comparative tests with some special methods which will be described in the latter portion of this report.

Twenty laboratories expressed a desire to take part in the cooperative work on potash samples, but reports were received from only eight laboratories.

Two samples were sent out for analysis to each laboratory taking part in the work, sample No. 1 being high-grade commercial sulphate of potash, while sample No. 2 was a mixed fertilizer, the ingredients of which were acid phosphate, cottonseed meal, dried blood, potassium chlorid, and a small amount of magnesium sulphate.

The following instructions with regard to the work were sent out to all cooperating chemists, the details of the volumetric method being those given by the referee for 1906 and 1907, Mr. A. L. Knisely, who had given much time and attention to a study of the phosphomolybdic method.

OUTLINE OF ASSOCIATION POTASH WORK.

Sample No. 1. Commercial sulphate of potash.

Sample No. 2. A complete mixed fertilizer, the nitrogen of which is derived from cottonseed meal and dried blood.

Potash in these samples should be determined both by the official method and the proposed volumetric method involving use of phosphomolybdic acid.

Reagents.

Nitric acid.—50 cc of nitric acid (1.40 sp. gr.) in 1,000 cc of water.

Sodium nitrate wash.—10 grams of sodium nitrate per 1,000 cc of water.

Phosphomolybdic acid solution.—100 grams of phosphomolybdic acid (Kahlbaum's preferred) in 750 cc of water and 250 cc of nitric acid (1.40 sp. gr.). This solution must be freshly prepared—not over three or four days old before using. If properly made the evaporated residue from a portion of this solution is never white and readily redissolves in the dilute nitric acid solution *in the cold*.

Standard solutions.—Standard caustic potash and nitric acid prepared for volumetric phosphoric acid diluted to 2 volumes. One cubic centimeter of this potassium hydroxid solution is equal to 0.812 mg of potassium oxid.

Determination.

Transfer 10 cc of solution to a platinum dish, add 0.25 cc of sulphuric acid (1 to 1). Evaporate to dryness and ignite to whiteness. Dissolve residue in hot water plus a few drops of hydrochloric acid and transfer to a tall 200 cc beaker, add 30 cc phosphomolybdic acid solution and slowly evaporate to complete dryness on top of a steam bath.

It requires approximately 22 mg of phosphomolybdic acid, in order to have an excess, for each milligram of potassium oxid present.

Add 30 cc of nitric acid wash to the dried residue and stir thoroughly in the cold, with a grinding motion with a policeman, allow to settle a moment and decant supernatant liquid at once through a gooch crucible packed with moist filter paper pulp, approximately one-sixteenth inch in thickness. Wash twice by decantation with sodium nitrate wash, transfer precipitate to a gooch and wash with sodium nitrate wash until acid free. Transfer gooch to casserole, run in excess standard alkali solution and add phenolphthalein. Heat to boiling and titrate excess alkali with standard acid.

Some samples of asbestos seem to hold or "fix" some of the excess acid, making the gooch filter very hard to wash acid free. Hence it is suggested to use a paper pulp filter. It is also desirable to make comparative tests, employing the usual asbestos filter.

If excess of phosphomolybdic acid has been used, the dried residue has a reddish hue. If excess has not been added the residue is bright yellow. Residue should not appear white.

In each case, run blanks to ascertain corrections to be made for impurities.

It is also desired that in sample No. 1 determinations of potash be made, not only by the official method (which provides for direct evaporation of the solution without addition of ammonia and ammonium oxalate), but also by the method applicable to mixed fertilizers, adding ammonia and ammonium oxalate, followed by evaporation and subsequent ignition with sulphuric acid.

Several chemists have urged that this latter method of procedure be tried, as it is claimed that the official method for potash salts gives too high results owing to impure precipitates.

The reports of results of cooperating chemists are as follows:

Potash results reported by cooperating chemists.

Analyst.	Sample No. 1			Sample No. 2.	
	Official method.	Volumetric method.	Official method plus ammonia and ammonium oxalate.	Official method.	Volumetric method.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
E. L. Baker, Geneva, N. Y.	51.32 51.24	51.26 51.30	50.16 50.16	4.41 4.43	4.89 4.72
S. E. Asbury, College Station, Tex.	51.40		50.66	4.42	4.65
E. C. Carlyle, College Station, Tex.					4.40
G. Farnham, Cincinnati, Ohio.	50.60		49.81	4.44	
G. Farnham, Cincinnati, Ohio.	50.56		49.77	4.40	
J. H. Mitchell, Clemson College, S. C.	50.80	50.41	50.64	4.44	4.45
B. F. Robertson, Clemson College, S. C.	50.75	50.60	50.62	4.47	4.49
G. T. Beyer, Chicago, Ill.	50.71	50.09	50.22	4.31	4.20
Laboratory of Armour & Co.	50.82 50.84	50.61 50.09	50.46 50.55	4.35 4.34	4.45
O. M. Shedd, Lexington, Ky.	50.68 50.73 50.29		50.16 50.08	4.38 4.43	
H. E. Taylor, Chicago, Ill.	51.02	58.19 53.90 61.60	50.28	4.42	4.66 4.68 5.01
Laboratory of Swift & Co.		58.00 57.96 59.62			5.14 4.99
C. R. Bragdon, Chicago, Ill.		58.80		4.43	4.90
Laboratory of Swift & Co.		59.11			4.69
C. L. Hare, Auburn, Ala.					4.30
A. M. Ransom and T. Bragg, Auburn, Ala.	51.20 51.36	51.96 52.60	49.80 50.10	4.44 4.38	4.60
Average	50.87		50.23	4.40	
M. G. Donk, Washington, D. C. ^b	50.92 50.80		49.06 49.00	4.38 4.35	4.21 4.32 4.41 4.36

^a 0.1 gram used.^b Results received too late to be included in the averages.

COMMENTS BY ANALYSTS.

E. L. Baker, Geneva, N. Y.: Moist filter paper pulp was used in one of each set of duplicates and a thick pad of asbestos in the other, with no appreciable variation in results. In some cases the precipitate showed a tendency to run through the filter paper pulp. It was easier, however, to wash the filter paper free from acid. Corrections were made for a blank of 0.3 cc of potassium hydroxid. Corrections were also made for blanks in the official method. You will notice that in the case of the mixed fertilizer the two methods differ by about 0.4 of a per cent. During a series of determinations I was unable to obtain any closer agreement.

E. C. Carlyle, College Station, Tex.: The use of pulped filter paper for filtering the phosphomolybdate is found satisfactory and it reduces the bumping when the liquid is heated for the purpose of dissolving the potash salt.

G. S. Farnham, Cincinnati, Ohio: I regret to report that I failed to get checks for the volumetric method.

P. Rudnick, Chicago, Ill.: It seems from the results by the official method that there is some truth in the claim that the method for mixed fertilizers when applied to sulphate of potash gives somewhat lower results. The proposed volumetric method

was given as thorough a trial as time and opportunity permitted, and although the results obtained were not very satisfactory, the method itself certainly looks very promising. The difficulties are, first, the very small amount of sample taken; second, the extreme proneness of the precipitate to go through the filter; third, the great difficulty of removing the precipitate from the sides of the beaker or casserole; fourth, the difficulty in washing all the nitric acid out of the precipitate. Asbestos is much inferior to paper pulp for filtering.

W. D. Richardson, Chicago, Ill.: With the volumetric method, following your directions, we did not have very good success.

O. M. Shedd, Lexington, Ky.: From my work, I would suggest on samples similar to No. 1 that ammonium hydroxid and ammonium oxalate be added as in the case of mixed fertilizers, and that an aliquot be used of 0.10 to 0.20 gram and not over 0.25 gram, instead of 0.50 gram, for the smaller potassium platonic chlorid precipitate can be worked better; besides it is my experience that very large precipitates carry down a greater proportion of impurities.

In addition to the above results Mr. Shedd made determinations in sample No. 1 by evaporating with sulphuric acid, igniting, and then evaporating with platonic chlorid. The results secured were 49.75 and 49.88 per cent.

M. G. Donk, Washington, D. C.: Could get no satisfactory results on sample No. 1 by the volumetric method.

E. L. Baker, associate referee, made additional comparative determinations of potash in several samples of potash salts, both with and without the use of ammonia and ammonium oxalate, in making up the solution, the results being as follows:

Comparison of results obtained with and without the use of ammonia and ammonium oxalate.

Sample.	With.	Without.	Sample.	With.	Without.
	<i>Per cent.</i>	<i>Per cent.</i>		<i>Per cent.</i>	<i>Per cent.</i>
Kalnit.....	12.94	13.13	Kalnit.....	13.42	13.48
	12.95	13.18		13.30	13.52
Muriate and sulphate.....	49.68	51.20	Muriate.....	49.12	50.04
	49.64	51.16		49.14	49.84

Mr. Baker also reported results of determinations of potash in a number of samples by the sodium cobalti-nitrite method first proposed as a quantitative process by Adie and Wood.^a This method involved the use of sodium cobalti-nitrite as a precipitant, and in the original process precipitation was effected in the presence of acetic acid in a solution which should contain from 0.5 to 1 per cent of potash. Drushel^b has modified this method as follows:

The solution of a potassium salt, containing not more than 0.2 gram of potassium oxid and free from ammonium salt, was treated with a rather large excess of sodium cobalti-nitrite solution, acidified with acetic acid, and evaporated to a pasty condition over the steam bath. It was then cooled and treated with from 50 to 100 cc of cold water, and stirred until the excess of sodium cobalti-nitrite was dissolved. It was allowed to settle and was decanted through a perforated crucible fitted with an asbestos felt. The precipitate was washed two or three times by decantation, after which it was transferred to the crucible and thoroughly washed with cold water. In the meantime a measured excess of standard potassium permanganate was diluted to ten times its volume and heated nearly to boiling. Into this the precipitate and felt were transferred and stirred, after which the crucible was also put into the solution, since particles of the precipitate stick persistently to its sides. After the oxidation had proceeded five or six minutes manganese hydroxid separated out and the

^aJ. Chem. Soc., 77: 1076.

^bAmer. J. Sci., 24: 433; Chem. News, 97: 124.

color of the solution darkened. At this point from 5 to 25 cc of sulphuric acid (1:2) were added, and the solution, after stirring, was allowed to stand a few minutes. Then a measured amount of standard oxalic acid, containing 50 cc of strong sulphuric acid per liter, was run in from a burette, taking care to add an excess. The temperature was maintained a little below the boiling point until the solution became colorless and the manganese hydroxid had completely dissolved. It was then titrated to color by permanganate in the usual manner. From the whole amount of permanganate employed, the permanganate equivalent of the oxalic acid used was subtracted and the remainder multiplied by the factor calculated for the strength of permanganate used, 0.000856 being the factor for strictly tenth-normal potassium permanganate.

While work was in progress in the referee's laboratory with a view to testing the adaptability of the cobalti-nitrite methods, letters were received from several cooperating chemists commending this process quite strongly, as a result of some preliminary work which had been done with it. Mr. A. M. Peter, of the Kentucky station, reported that by the cobalti-nitrite method results of 49.89 and 49.92 per cent were obtained by Mr. G. Edgar for sample No. 1, as against 49.82 by the official method, while for sample No. 2, 4.50 and 4.48 per cent of potash was found, as against 4.41 by the official method. Mr. Baker obtained the following results on the official samples by Drushel's modification: No. 1, 50.24 and 50.85 per cent; No. 2, 4.46 and 4.31 per cent. Following are results reported by Mr. Baker, using the original Adie and Wood method, in which precipitation is effected without evaporation:

Comparison of potash determinations by the original Adie and Wood cobalti-nitrite method (volumetric) and the official method.

Sample.	Official method.	Cobalti-nitrite method.	Sample.	Official method.	Cobalti-nitrite method.
	<i>Per cent.</i>	<i>Per cent.</i>		<i>Per cent.</i>	<i>Per cent.</i>
Sulphate.....	53.54	54.08	Kainit a.....	13.13	10.14
	53.70	54.08		13.18	10.28
Muriate and sulphate.....	50.68	50.71	Mixed fertilizer a.....	4.95	4.21
	50.84	50.71		5.06	4.21
Mixed fertilizer a.....	10.41	10.78	Mixed fertilizer a.....	1.28	.40
	10.41	10.92		1.29	.40
Kainit a.....	13.48	10.64			
	13.52	10.71			

a The kainits and mixed fertilizers evidently did not entirely precipitate owing, probably, to improper concentration.

CONCLUSIONS.

It appears from the result of this year's work, that while some good results are obtained by the volumetric method, there are difficulties connected with the working of the process which affect the reliability and rapidity of its execution. Among these may be mentioned the trouble experienced in washing the precipitate free of acid and the tendency of the precipitate to run through, while the smallness of the aliquot used in the determinations would, of course, tend to affect the accuracy of the results.

On this account it would seem desirable that work with this method be held in abeyance for the present and that a trial be made of the cobalti-nitrite method with a view to determining its adaptability to fertilizer work.

The results of tests of the employment of ammonia and ammonium oxalate in potash determinations in potash salts indicated that lower figures are secured in this way, so that from this partial investigation the contention of those who claim that the usual method gives impure precipitates would seem to be sustained. However, no positive conclusion can be reached from the limited data at hand and hence this question should be investigated further.

REPORT OF COMMITTEE C (FOOD ADULTERATION).By L. M. TOLMAN, *Chairman.***WINES.**

It is recommended—

(1) That a committee of five be appointed to cooperate with the Bureau of Standards in drawing up a standard alcohol table.

Adopted.

(2) That the question of a standard temperature of 20° for specific gravity and alcohol determinations be also referred to the committee of five.

Adopted.

(3) That the following subjects be given further study:

(a) Methods for determining glycerol.

(b) Methods for determining total, fixed, and volatile acids.

(c) Methods for determining coloring matter in genuine wines.

Adopted.

FLAVORING EXTRACTS.

It is recommended—

That the colorimetric method for the determination of citral in lemon extract be adopted as provisional. (See page 32.)

Adopted.

DAIRY PRODUCTS.

It is recommended—

That the Baier and Neumann method for the detection of sucrose of lime in milk and cream be studied. (See page 53.)

Adopted.

DISTILLED LIQUORS.

It is recommended—

(1) That the modified Allen-Marquardt method for the determination of fusel oil be made a provisional method.

Adopted.

(2) That in the present method (Bul. 107, Rev., p. 98) a second washing with sodium sulphate be prescribed.

Adopted.

(3) That the method for the determination of water-insoluble color in whiskies be made provisional. (See page 207.)

Adopted.

(4) That the modified Marsh test for the quantitative determination of the color insoluble in amyl alcohol be adopted as a provisional method. (See page 206.)

Adopted.

(5) That the provisional Roese method for determining fusel oil (Bul. 107, Rev., p. 97) be dropped.

Adopted.

SPICES.

It is recommended—

That methods for the detection of added oil in paprika be further studied.

Adopted.

MEAT AND FISH.

It is recommended—

(1) That the study be continued in an attempt to apply, improve, or devise methods for the most accurate separation possible of the various protein bodies in meat.

(2) That the method for determining ammoniacal nitrogen by distillation under reduced pressure be compared with the magnesium oxid method now generally used. (Bul. 107, p. 9.)

CEREAL PRODUCTS.

It is recommended—

That methods applicable for the separation of the gluten constituents of flour be studied, tests to be made upon the several grades, as patents, first and second clears, and on flours produced from different varieties and types of wheat.

Adopted.

CANNED VEGETABLES.

It is recommended—

That methods for the detection of soaked peas be further studied.

Adopted.

MEAT PROTEIDS.

It is recommended—

That the work on the separation of meat proteids be continued along the lines pursued the past year.

Adopted.

TEA, COFFEE, AND COCOA.

It is recommended—

(1) That methods for the estimation of caffein be further studied. (Original Gomberg Method, *J. Amer. Chem. Soc.*, 1896, p. 331, and modifications.)

Adopted.

(2) That the Dubois method for the determination of sugars in chocolate be further studied. (*J. Amer. Chem. Soc.*, 1907, 29: 556; *Bul.* 107, p. 256.)

(3) That the Doolittle and Woodruff method (*Bul.* 105, p. 48) for extract in tea be substituted for the Krauch method (*Bul.* 107, p. 149, sec. 5) as provisional.

Adopted.

COLORS.

It is recommended—

(1) That an effort be made to obtain authentic samples of vegetable or natural coloring matters, such as are used in food products.

(2) That a study be made of the characteristics of vegetable coloring matters and methods of identification.

(3) That pure colors be synthetically prepared to serve as standards.

(4) That the separation and identification of mixed colors be studied.

These recommendations were adopted.

REPORT OF COMMITTEE ON THE TESTING OF CHEMICAL REAGENTS.

By L. F. KEBLER, *Chairman.*

There has been a marked improvement in the chemical reagents examined by the chairman of the committee during the past year. This, however, may be largely due to a weeding-out process that has been going on for several years. It was a common experience a few years ago to be compelled to report adversely on the quality of many chemicals which included not only actual adulteration, but indicated gross carelessness in manufacturing and packing. The chemicals found to be of inferior quality during the past year were generally lacking in certain minor respects; for example, contamination with insoluble material or some associated impurity which would be detrimental to the analytical operations for which the reagent was to be employed.

One of the difficult features at present is a satisfactory nomenclature. In the past it has been common to use in connection with chemicals supposed to be of high quality the abbreviation C. P., but this abbreviation has come to be meaningless and should be discontinued. It still serves one good purpose and that is, if a chemical

is accompanied by this designation the chemist can reject it on general principles if found to be of unsatisfactory quality. Other specifications, such as pure, purissimum, reagent, commercial, etc., also have vague meanings which are used by manufacturers, dealers, and brokers, simply as a means for selling certain chemicals. The past year has seen a marked improvement along these lines, due largely to the instrumentality of the food and drugs act. The term "commercial" has been replaced largely by the term "technical" for the reason that the former name was vague and was used in connection with products which might be used for either food, drug, or technical purposes; for example, "sodium phosphate, commercial," did not give any information at all as to the quality of the product, and while the name would suggest that it was not of high grade, yet it was not uncommon for highly arsenical sodium phosphate to find its way into the drug trade, rather than to the boiler compound manufactory, and thus do harm. The terms pure, purissimum, and reagent are also gradually losing their standing, and the question arises, What form of nomenclature should be employed in order to obtain chemicals of the desired quality?

The chairman, therefore, recommends that the committee be instructed to investigate the question of nomenclature to be used in connection with chemical reagents and report at the next meeting.

The report was accepted and the recommendation made was approved by the association.

REPORT OF COMMITTEE ON FOOD STANDARDS.

On behalf of the food standards committee of the association, the chairman, Mr. Frear, submitted a detailed report of the work done by the joint committee on food standards during the year. This covers the adoption of tentative standards for manufactured meats, malt liquors, and spirituous liquors. The report of the committee was accepted by the association.

The president announced the following committee on the standardization of alcohol tables: L. M. Tolman, M. E. Jaffa, A. B. Adams, R. J. Davidson, H. E. Barnard.

REPORT OF COMMITTEE ON NOMINATIONS.

Mr. Davidson, as chairman of the committee on nominations, then presented the following report: For president, Mr. W. D. Bigelow; for vice-president, Mr. W. A. Withers; for secretary, Mr. H. W. Wiley; for additional members of the executive committee, Mr. E. F. Ladd and Mr. E. B. Holland.

The chairman of the committee was instructed to cast the unanimous vote of the association for the officers named.

On motion by Mr. Davidson the question of the amount of wash water to be employed in the treatment of the residue from the ammonium citrate digestion in the determination of phosphoric acid was referred to Committee A for recommendation.

THE ASSAYING OF ALKALOIDAL DRUGS.

By C. E. PARKER.

The original drug assay methods of the last revision of the United States Pharmacopœia, on the whole, fairly represented the existing status of this branch of chemical analysis. They were formulated under the instruction of the convention for revising the Pharmacopœia that assay processes should be "reasonably simple (both as to methods and apparatus required) and lead to fairly uniform results in different hands."

The probability being somewhat vague that they would be made the basis for general legal regulation, a high degree of accuracy did not appear important, and similar moderate standards of requirement have possibly influenced the evolution of drug assay methods generally. After the passage of the federal food and drugs act of June 30, 1906, the committee on revision made a number of corrections and modifications in the text of the Pharmacopœia that it might better meet the new requirements.

Judged from the point of view of the official chemist and prospective expert witness before the courts, the cooperative work as far as it has gone has not shown that the pharmacopœial methods lead to fairly uniform results in different hands. This is probably due more to lack of detail in the instructions than to any fundamental defects in the methods. It is evident that losses occurring at certain stages in the processes may be prevented by suitable alterations in the methods, and that the unfavorable results on some drug samples may, to a considerable extent, be attributed to the powder not being of a proper fineness.

The samples sent out this year were from supplies ordered to be according to the United States Pharmacopœia, both as to assay and fineness of powder. The sample of belladonna root has been criticised as being a finer powder than specified by the Pharmacopœia, and, therefore, likely to give higher results and too favorable reports on the method. Other samples of drugs have been said to be too coarse, and, therefore, unfair to the methods. The point is well taken, but the only way to obtain a powder of exactly the pharmacopœial size would be to separate with suitable screens all larger and smaller particles produced by the mill, and such a product would not be representative of the original drug. The proper solution of the difficulty would seem to be the provision of suitable apparatus for grinding all drug samples for assay at least as fine as the Pharmacopœia requires and as much finer as experience shall show to be expedient.

The theoretical objections to the aliquot method of extraction may be justified when the grosser imperfections in the methods have been eliminated, but so far results fail to demonstrate the superior reliability of the total extraction method, and judgment must be suspended.

It was thought advisable to traverse again the ground covered last year when only three analysts participated, comprising methods for the assay of aconite root, belladonna leaves, belladonna root, cinchona bark (yellow and red), cocoa leaves, colchicum corm, and colchicum seeds. Samples of these drugs delivered as being of pharmacopœial quality and as ground to the fineness of powder specified in the respective pharmacopœial assay methods were supplied to all collaborators with the following directions, and instructions that all calculations and solutions except as otherwise specified be based on the data of the United States Pharmacopœia, eighth revision, with the additions and corrections dated May 1 and June 1, 1907.

The provisional methods appearing in Bulletin 107, revised, pages 258-259, were slightly modified in accordance with the experience of last year. *Only the modifications are reprinted below and the changes are italicized.*

DETERMINATION OF ALKALOID.

Total extraction method.

Into a 200 cc flask weigh 10 grams of the powdered drug, add about 75 cc of ether-chloroform mixture (5 to 1 by volume), rotate and add 5 cc of 10 per cent ammonia water, cork, shake well and often during two hours. * * *

Aliquot method.

Into a 200 cc flask weigh 15 grams of the powdered drug, add 150 cc of ether-chloroform mixture (5 to 1 by volume), cork and shake often for several minutes. Add 5 cc of ammonia water (10 per cent), shake frequently during two hours. Add 15 cc of water, or sufficient to agglomerate the drug, shake, let settle a few minutes, and then decant 100 cc of the clear solution into a graduated cylinder. * * *

NOTE.—Under both methods substitute “a few cubic centimeters” for the words “a small portion,” referring to the ether-chloroform rinsing.

CINCHONA BARK.

Method I.

United States Pharmacopœia VIII, page 102. Report total and ether-soluble alkaloids.

Method II.

Total extraction, gravimetric. *In extracting the drug let stand over night.*

The work on yellow and red cinchona and colchicum corm and root being quite incomplete is not included in this report. The instructions should be followed as strictly as possible, notes being taken during the work of any difficulties encountered, objections to the methods, necessary or advisable modifications with the reasons therefor, and any ambiguity or indefiniteness in the instructions should be indicated. The value of collaborators' reports is much enhanced by this practice. (See tabulation at close of report, p. 134.)

For comparing in respect to their variability the results obtained by the different methods from the several drugs, the average result for each method is taken as a basis, and the proportion of all the results approaching within 10 per cent above or below this average is given, and in addition the proportion approaching within 15 per cent of the average. Reserving the question of absolute accuracy, results commonly varying over a range of more than 20 per cent in different hands can scarcely be described as fairly uniform, nor can methods yielding such results be considered satisfactory for the purposes of the official chemist. Only one operator has reported any dissatisfaction with the behavior of cochineal as an indicator, though another has substituted hematoxylin for it throughout.

DETAILS OF MANIPULATION.

The United States Pharmacopœia assay methods generally direct that the initial digestion of the drug with a solvent for the purpose of extracting the active principle be accompanied by an indefinite amount of agitation. In certain cases continuous agitation by means of suitable mechanism is alternatively directed, or preferred. The expression “frequent shaking” is susceptible of various interpretations, and it would be advisable to adopt the requirement of continuous agitation in all cases.

A number of collaborators reported difficulty in decanting 100 cc of the solvent mixture in extracting the drug by the aliquot method, and some were compelled to use forcible expression or continue the assay with less than 100 cc, computing the result on the basis of the aliquot part decanted. This occurred especially with belladonna leaves and cocoa leaves and is attributable to the coarseness of the samples. In the Drug Division it was found practicable to obtain 100 cc by decanting the mixture of drug and solvent as completely as possible into a small percolator provided

with a purified cotton plug in the neck, and loosely stoppering the same while the filtrate collected in a 100 cc flask. Excessive evaporation was thus avoided. With samples of a suitable degree of fineness 100 cc could be decanted without difficulty.

One worker filters the final solution of alkaloid in volatile solvent before evaporating off the latter. If the funnel be kept covered during filtration, and if the filter be properly washed, losses may be avoided and the alkaloid obtained in a cleaner condition than without filtration.

DISCUSSION OF RESULTS.

ACONITE ROOT.

This sample was delivered as No. 40 powder. The following proportions passed through the respective sieves:

	Grams.
No. 60.....	0
No. 50.....	7
No. 40.....	11
No. 20.....	82
Total.....	100

Most of the powder was therefore coarser than the Pharmacopœia directs for assay samples.

The three gravimetric results by Method I are too few in number to base upon them any conclusion. Only 32 per cent of the volumetric results by Method I (U. S. P.) come within 10 per cent of the average and only 59 per cent come within 15 per cent, and the results, both gravimetric and volumetric, by (II) are, on the whole, as bad or worse. The average results by the two methods are in very good agreement, but considerably under the United States Pharmacopœia standard of 0.50 per cent. It is quite possible that higher and more uniform results might have been obtained with a finely powdered sample.

On comparison of the corresponding gravimetric and volumetric results by (II) which we may assume were obtained by weighing and then titrating the same alkaloidal residues, it will be observed that in about one-half the instances the volumetric result is higher than the gravimetric, though it can not be assumed that these residues consist of absolutely pure alkaloid. The factor for aconitine (0.064) employed in computing the volumetric result is too high, and the residue contains alkaloidal matter of lower molecular weight than 640, resulting from the decomposition of aconitin. It is probable that the volumetric results by (I) are affected by a similar error. These considerations tend to support the contention of Doctor Lyons and others that chemical assays of aconite should be confirmed by the so-called "physiological test."

In Method, I Mr. Fuller considers the evaporation of the alcoholic percolate to dryness at a temperature not exceeding 60° as too tedious, and carried evaporation only to the point where alcohol was all expelled, acidifying the aqueous residue with normal acid and filtering as usual. He also washed the acid solution with ether before making alkaline and shaking out. A number of workers note the usual difficulty in filtering the acidified residue from evaporation. Mr. Hankey added powdered pumice to the residue to aid filtration and titrated finally with half-strength lime water. He found the marc on repeating the extraction yielded no more alkaloid. Mr. La Wall in a parallel experiment shook out finally with chloroform-ether mixture instead of ether and obtained lower results, viz, gravimetric 0.35 per cent and volumetric 0.416 per cent. Doctor Lyons used paper pulp to aid filtration, and after the final shaking out with ether further shaking out with chloroform yielded about 0.1 per cent alkaloid, titrating 0.075 per cent as aconitin and producing its characteristic effect on the tongue. He believes that aconite assays should be confirmed by the Squibb physiological test. He also suggests a direct titration method for aconite, similar to the

United States Pharmacopœia method for belladonna in the details of extracting the drug, but instead of shaking out the ethereal extract with acid, the former is to be evaporated, ammonia expelled by repeated addition of a few cubic centimeters of ether and evaporation, and the impure residue titrated. It might either be dissolved in alcohol diluted with water and titrated with acid, or dissolved in excess of standard acid and the excess of acid titrated with standard alkali, preferably with iodeosin indicator. This method, he thinks, could be adapted for many alkaloidal drugs. Professor Ruddiman criticises the use of decinormal acid, especially in titrating an alkaloid of such high molecular weight as aconitin, where a slight difference in measurement seriously affects the result.

In Method II as well as in I, Mr. Lyons obtained a further yield of about 0.1 per cent of alkaloid by shaking out with chloroform following the final extraction of the alkaline liquid with ether. Mr. Pearson redissolved the alkaloidal residues from the gravimetric determinations in (II) in acid and purified by submitting them to a shaking-out process with ether, obtaining much lower results, viz, 0.312 and 0.315 per cent.

In view of the fact that both methods gave practically the same average volumetric result and variability, the greater convenience and rapidity of Method II are in its favor.

BELLADONNA LEAVES.

This sample was delivered as No. 60 powder. The following proportions passed through the respective sieves:

	Grams.
No. 60.....	40
No. 50.....	35
No. 40.....	25
Total.....	100

A considerable amount of coarser powder than the Pharmacopœia permits in assay samples of belladonna leaves was present. By Method I (U.S.P.) the few gravimetric results reported varied exceedingly, none of them coming within 10 per cent of the average, and only 14 per cent within 15 per cent of the average. Of the volumetric results, 41 per cent came within 10 per cent and 65 per cent within 15 per cent of the average. By (II) gravimetric, 86 per cent of the few results were within 10 per cent; also 86 per cent within 15 per cent of the average. Of the volumetric results by (II) 39 per cent came within 10 per cent, and 73 per cent within 15 per cent of the average. The average results by (I) are slightly higher than by (II), but both are somewhat under the United States Pharmacopœia standard of 0.30 per cent. A slight impurity in the residues is indicated by the higher gravimetric results. In (I) Mr. Hankey used 2 cc of ether to assist solution of the alkaloidal residue in acid, expelling it by gentle warming before titration. J. G. Francis and Parker used 50 cc more ether-chloroform mixture than directed to exhaust the drug. It has been observed in the Drug Division when assaying belladonna leaves and root and coca leaves by the pharmacopœial method^a that a large portion of the last 50 cc of solvent mixture which is intended to complete the percolation has to be used in rinsing the drug into the percolator. The drug should be packed after it is all transferred and percolation carried to practical exhaustion. The combined acid solutions obtained by shaking with the percolate should be shaken with fresh solvent in small portions until no more color is removed before making alkaline and shaking out the alkaloid. Instead of measuring out 3 cc of decinormal sulphuric acid to dissolve the alkaloid, a number of workers in such

^a Workers in the Division of Drugs recommend cylindrical nursing bottles (8 ounces) which taper to the neck without any shoulder instead of Erlenmeyer flasks for digesting the drug with solvent, as the former are more easily clamped on a mechanical shaker.

cases prefer to add an equivalent amount of fiftieth-normal acid as a quantity less liable to error in measurement.

In (II) Mr. Blome suggests increasing the amount of ether-chloroform mixture for extracting the drug to 180 cc and decanting 120 cc. Mr. Fuller suggests that instead of directing the use of neutral alcohol for dissolving the alkaloid before titration it would be preferable to compare the result with that of a blank titration made with the same amount of the same stock of alcohol, water, and indicator. Mr. Hankey reports dissatisfaction with the titration results owing to an indefinite end reaction. Though his alcohol was redistilled over alkali, a blank titration with the amounts of acid, alcohol, and water directed required only 14.3 cc of fiftieth-normal alkali, while the same amount of acid by direct titration required 15 cc of the standard alkali. Mr. Parker prepared "neutral" alcohol by adding fiftieth-normal potassium hydroxid to alcohol until a blank titration with the amounts of acid, alcohol, and water directed agreed with a direct titration of the acid alone. This method or that suggested by Mr. Fuller eliminates the effect of any deviation from neutrality by the alcohol or water under the working conditions. Mr. Lyons made a parallel experiment, evaporating the ether-chloroform extract of the drug instead of shaking out with acid and titrating the residue directly, as outlined in the discussion under aconite root. The result was 0.32 per cent.

BELLADONNA ROOT.

This sample was delivered as No. 60 powder, and passed through the several sieves in the following proportions:

	Grams.
No. 80.....	98
No. 60.....	1
Total.....	99

It was, therefore, somewhat finer than the Pharmacopœia requires for assay samples of this drug. By Method I (U. S. P.) of the few gravimetric results 29 per cent came within 10 per cent of the average and 43 per cent within 15 per cent. Of the volumetric results, 46 per cent came within 10 per cent of the average and 80 per cent within 15 per cent. By (II) the gravimetric results varied more than the similar determinations by (I). The volumetric results by (II) were decidedly better than the corresponding results by (I), 73 per cent coming within 10 per cent of the average and 85 per cent within 15 per cent. The averages by the two volumetric determinations are practically identical, likewise those by the two gravimetric determinations, but no explanation is apparent for the fact that by both methods the gravimetric results average lower than the volumetric. This relation occurs also in four instances (in II) where the results apparently represent the same residue.

In (I) Mr. Hankey dissolved the alkaloidal residue in 1 cc of neutral alcohol before adding excess of standard acid and titrating back with half-strength limewater, comparing the same with a blank titration. C. H. La Wall made parallel assays by both methods, evaporating the ether-chloroform extract instead of shaking out with acid, and titrating the impure residue directly, the results obtained being (I) 0.514 and (II) 0.529 per cent, duplicate results agreeing well. J. G. Francis used 25 cc, and Mr. Parker 50 cc more ether-chloroform mixture than the amount directed to extract the drug. Their results are all well above the average. The remarks made in the discussion on belladonna leaves, Method I, regarding the percolation of the drug also apply to belladonna root. With belladonna root Method II, by evaporation of the ether-chloroform extract, and direct titration of the impure residue, Mr. Lyons obtained a value of 0.617 per cent.

Cooperative work on assaying alkaloidal drugs.

Collaborator.	Aconite root.				Belladonna leaves.				Belladonna root.				Coca leaves.			
	Method I (U. S. P.).		Method II.		Method I (U. S. P.).		Method II.		Method I (U. S. P.).		Method II.		Method I (U. S. P.).		Method II.	
	Grav.	Vol.	Grav.	Vol.	Grav.	Vol.	Grav.	Vol.	Grav.	Vol.	Grav.	Vol.	Grav.	Vol.	Grav.	Vol.
Blome, W. H.	Per ct. 0.385	Per cent. 0.317	Per cent. 0.280	Per cent. 0.294	Per cent. 0.280	Per cent. 0.294	Per cent. 0.280	Per cent. 0.294	Per cent. 0.280	Per cent. 0.294	Per cent. 0.280	Per cent. 0.294	Per cent. 0.280	Per cent. 0.294	Per cent. 0.280	Per cent. 0.294
Darling, J. F.	a. 384	a. 310	a. 326	a. 270	a. 326	a. 270	a. 326	a. 270	a. 326	a. 270	a. 326	a. 270	a. 326	a. 270	a. 326	a. 270
Faton, E. O.	a. 308	a. 315	a. 330	a. 283	a. 337	a. 283	a. 337	a. 283	a. 337	a. 283	a. 337	a. 283	a. 337	a. 283	a. 337	a. 283
Francis, J. G.	a. 411	a. 465	a. 480	a. 424	a. 329	a. 424	a. 329	a. 424	a. 329	a. 424	a. 329	a. 424	a. 329	a. 424	a. 329	a. 424
Fuller, H. C.	a. 436	a. 450	a. 462	a. 316	a. 350	a. 316	a. 350	a. 316	a. 350	a. 316	a. 350	a. 316	a. 350	a. 316	a. 350	a. 316
Hankey, W. T.	a. 436	a. 366	a. 353	a. 316	a. 310	a. 316	a. 310	a. 316	a. 310	a. 316	a. 310	a. 316	a. 310	a. 316	a. 310	a. 316
Havenhill, L. D.	a. 420	a. 348	a. 350	a. 313	a. 327	a. 313	a. 327	a. 313	a. 327	a. 313	a. 327	a. 313	a. 327	a. 313	a. 327	a. 313
Kirschmaier, G. A.	a. 218	a. 295	a. 350	a. 276	a. 276	a. 276	a. 276	a. 276	a. 276	a. 276	a. 276	a. 276	a. 276	a. 276	a. 276	a. 276
La Wall, C. H.	a. 390	a. 448	a. 370	a. 441	a. 212	a. 220	a. 212	a. 220	a. 212	a. 220	a. 212	a. 220	a. 212	a. 220	a. 212	a. 220
Lilly, Eli, & Co.	a. 474	a. 371	a. 315	a. 435	a. 324	a. 315	a. 324	a. 315	a. 324	a. 315	a. 324	a. 315	a. 324	a. 315	a. 324	a. 315
Lyons, A. B.	a. 400	a. 435	a. 450	a. 300	a. 250	a. 300	a. 250	a. 300	a. 250	a. 300	a. 250	a. 300	a. 250	a. 300	a. 250	a. 300
Nelson, B. E.	a. 578	a. 783	a. 512	a. 388	a. 230	a. 313	a. 230	a. 313	a. 230	a. 313	a. 230	a. 313	a. 230	a. 313	a. 230	a. 313
Parker, C. E.	a. 435	a. 732	a. 812	a. 420	a. 215	a. 325	a. 215	a. 325	a. 215	a. 325	a. 215	a. 325	a. 215	a. 325	a. 215	a. 325
Pearson, W. A.	a. 422	a. 288	a. 273	a. 339	a. 339	a. 347	a. 339	a. 347	a. 339	a. 347	a. 339	a. 347	a. 339	a. 347	a. 339	a. 347
Ruddiman, F. A.	a. 378	a. 725	a. 384	a. 318	a. 318	a. 318	a. 318	a. 318	a. 318	a. 318	a. 318	a. 318	a. 318	a. 318	a. 318	a. 318
	a. 384	a. 765	a. 384	a. 310	a. 290	a. 310	a. 290	a. 310	a. 290	a. 310	a. 290	a. 310	a. 290	a. 310	a. 290	a. 310
	a. 330	a. 403	a. 330	a. 290	a. 160	a. 290	a. 160	a. 290	a. 160	a. 290	a. 160	a. 290	a. 160	a. 290	a. 160	a. 290
	a. 394	a. 389	a. 394	a. 140	a. 140	a. 140	a. 140	a. 140	a. 140	a. 140	a. 140	a. 140	a. 140	a. 140	a. 140	a. 140
	a. 333	a. 403	a. 333	a. 160	a. 160	a. 160	a. 160	a. 160	a. 160	a. 160	a. 160	a. 160	a. 160	a. 160	a. 160	a. 160

COCA LEAVES.

This sample, delivered as No. 60 powder, passed through the several sieves in the following proportions:

	Grams.
No. 60.....	29
No. 50.....	18
No. 40.....	9
No. 20.....	43
Total.....	99

A large portion of the powder was coarser than the pharmacopœial requirement for assay samples of this drug. The gravimetric results by Method I (U. S. P.) are too few to justify any conclusions. Of the volumetric results, 36 per cent come within 10 per cent and 58 per cent within 15 per cent of the average. Of the gravimetric results by (II), 75 per cent come within 10 per cent and 82 per cent within 15 per cent of the average. Of the volumetric results, 33 per cent come within 10 per cent and 72 per cent within 15 per cent of the average. The gravimetric averages and likewise the volumetric averages by the respective methods are in substantial agreement, the gravimetric results being somewhat higher than the volumetric, owing probably to impurities in the alkaloidal residue. In (I) Mr. Fuller accomplished the final shaking out with three portions of 20 cc each of ether instead of 25, 20, and 15 cc. He thinks the drug should be digested with the solvent mixture longer than one hour, as the marc in this case still contained alkaloid. Mr. Hankey dissolved the alkaloidal residue with 1 cc of "neutral spirits" and titrated with acid and diluted limewater as with belladonna root. Cochineal gave an unsatisfactory end reaction. Messrs. La Wall and Parker noted considerable emulsification in shaking out by both methods. The latter used 50 cc more solvent than is directed for percolating the drug, and J. G. Francis used 75 cc more, and shook the drug finally with five portions of 25 cc of ether. The extraction was not complete.

For coca as for belladonna the amount of solvent mixture directed in the United States Pharmacopœia method is scarcely adequate for the proper manipulation and extraction of the drug. In the final shaking out process further extraction with ether is desirable. In (II) Mr. Blome suggests increasing the ether-chloroform mixture to 180 cc and decanting 120 cc. Mr. Hankey obtained a better end reaction with iodeosin than with cochineal. Professor La Wall obtained equally low results in a duplicate assay. J. G. Francis found that the final extraction was not complete. Mr. Pearson could not decant 100 cc without forcible expression, and therefore objects to the method. As in (I), further extraction with ether in the final shaking out is probably desirable.

In both (I) and (II) considerable impurity evidently passes into the alkaloidal residue, and a more thorough washing with solvent before making alkaline is indicated.

THE MACROSCOPY AND MICROSCOPY OF DRUGS.

By H. H. Rusby.

The object of this brief paper is to direct the attention of the members to the importance of chemists supplementing their chemical methods by suitable physical methods in identifying and estimating drugs; and to the facility with which the chemist can acquire enough knowledge of such physical methods, and of the physical properties of drugs, to be of great assistance in his analytical work.

When the subject of the chemical standardization of vegetable drugs was being agitated in connection with the approaching United States Pharmacopœia Convention of 1890, the writer was astonished to hear Prof. John M. Maisch declare himself

opposed to the introduction of such standards into the Pharmacopœia. This surprise was considerably augmented when Doctor Maisch gave as his reason the statement that if a man knew drugs as he should it would not be necessary to examine them chemically to determine their quality. Although we can not in these days admit the propriety of neglecting chemical standardization, for this or any other reason, yet subsequent experience has shown that Doctor Maisch's claim to be able to judge the quality of drugs without recourse to chemical methods is largely justified.

The necessity of such knowledge is apparent when we reflect that of the 167 crude vegetable drugs of the Pharmacopœia, chemical standards are prescribed for only 22, and yet the Pharmacopœia does not recognize more than one-half of the nonstandardized articles in common use. It is true that chemists employ quantitative methods, all more or less satisfactory, in the case of ten or a dozen others, which are not thus treated in the Pharmacopœia. Admitting these to full membership, how overwhelming still is the majority upon the other side! Let it not be said that the non-assayable list represents only unimportant drugs. It is one of the great temptations of the chemist to underrate subjects with which he does not deal, and he is apt to reason *post hoc, ergo propter hoc*. Let us not forget that it is the extreme variability in activity of such drugs as veratrum, digitalis, ergot, and cannabis indica, coupled with their exceeding importance in medicine, which has forced a resort to physiological standardization, applicable as yet to but few drugs. It is this tendency to vary in quality and our general inability to estimate such quality that has to a great extent destroyed the usefulness of some drugs which would otherwise be generally relied upon. As illustrations, let us note male fern, spigelia, cuso, and other anthelmintics, Winter's bark, coto bark, and chrysarobin. The importance of the drugs named is relatively greater than that of the assayable ones, by virtue of the fact that the latter can be substituted by their proximate principles, while the former can not.

There is yet another element of weakness in the chemical assay of drugs, which is greatly mitigated by attention to their macroscopical and microscopical characters. Every assayer is frequently more or less chagrined by the thought that after all he does not know what it is that he has in hand after he has extracted the full required percentage of alkaloid by the prescribed method, since part of it may have been extracted from an admixture. Impurities in drugs, either from accident or design, may and frequently do fail of detection by the chemist, even in the case of freely assayable drugs, where detection would be simple by intelligent physical examination before assaying.

Even the great array of unofficial and unimportant drugs can not be dismissed from the chemist's ken because of their want of substantial therapeutic activity. They are in common use and some one pays for them the money which is his property and which entitles him to the receipt of what he pays for. He may be deprived of the protective aid of the Pharmacopœia without having his legal or professional rights in any degree curtailed. Indeed, the chemist himself is a deeply interested party in this class of transactions. Every commercial chemist will admit that some of his most profitable work lies in the field of the unofficial *materia medica*, and where the distinctly chemical indications are usually indefinite and faint. It seems quite unnecessary to argue further that a knowledge of the physical identification characters of vegetable drugs is of great service to the chemist. Is it too much to say that the field of success thus opened to him is far greater, as to crude vegetable drugs, than that which he can control by chemical methods alone? I feel very sure that such a statement is just and moderate.

This being so, how far can macroscopical and microscopical methods supply the deficiency? And how great an expenditure of effort and time does it require? It may be admitted at once that to secure an expert knowledge of this subject requires the same kind and degree of application that it does to become an expert chemist, but it is at the same time true that a very moderate amount of effort, intelligently

and judiciously applied, will add more to the general efficiency of the chemist than the same amount applied in any other direction. I believe that no chemist should proceed with the chemical examination of a drug of this class until after he has examined it physically, with or without the microscope, according to the requirements of the case, to ascertain its general characters and particularly whether it is a single article or a mixture. This requires a fair knowledge of macroscopy and microscopy, as to both methods and drugs. The time and labor necessary to acquire such a knowledge are not excessive. As to all the official and important unofficial drugs, it should be gained by from one hundred to one hundred and fifty hours of practical work, say two or three hours per week during a two-year course.

The following examples will serve to illustrate the class of drugs to which reference is here made: Coto and paracoto bark are among the most reliable therapeutic agents in the *materia medica*, often the only means of saving life in severe cases of dysentery, yet the use of this medicine has almost ceased owing to the fact that the genuine drug is now scarcely ever seen. In two years the writer has not known of an importation of it to the United States that was not spurious. A brief macroscopic examination will enable anyone immediately to recognize every one of these pretenders. The same statement applies, in a somewhat less serious degree, to Winter's bark, a most valuable aid in nutrition.

The belladonna invoice covers a multitude of fatal and dangerous imperfections. A very large part of our belladonna root contains poke root, not only an exceedingly active poison but an article that counteracts the medicinal effect of belladonna. It is sometimes difficult to distinguish the smaller roots by macroscopical means, but the dust in the package will always show, under the microscope, the needle-shaped crystals of the poke root. The same statement applies to an admixture of poke leaves to belladonna leaves. Scopolia leaves are often mixed with and substituted for belladonna leaves. This is liable to destroy the life of the patient receiving the medicine. In any case the medicinal actions of these two are antagonistic. Some indication of the identity of these plants is almost always present with the leaves; for example, the belladonna has black berries, while the scopolia has pale yellow circumscissile pods, and the two can be instantly distinguished.

A spurious henbane sometimes contains from ten to fifteen times as much alkaloidal matter as the genuine and has a different action. These alkaloids are so poisonous that they are given in doses of only one two-hundred-and-fiftieth to one one-hundredth of a grain. Imagine the effect of giving a dose containing fifteen times as much as it should. When powdered, the spurious can be recognized by its stellate hairs and by certain cells with wavy thick walls. Henbane and digitalis may contain stramonium leaves. Any considerable amount of such an addition to digitalis must put the life of the patient in danger, because with heart failure life often depends upon the full and prompt action of the latter remedy. Here the microscope is almost necessary, as a single hair from the leaf of the stramonium, densely covered with minute warts, will tell the story.

Strophanthus seed is another drug of great service in heart failure, and used when promptness is necessary. There is one variety of the seed which produces no good effect, and there has been ten times as much of this used in the United States as of the other, because it has cost only one-tenth to one-fifth as much. During the past year the use of the spurious kind has been largely stopped. The two seeds have such different macroscopic characteristics that they can not be mistaken when once the difference has been noted.

So-called saffron is frequently found which consists of marigold flowers, colored red with anilin and heavily weighted with mineral matter. The evil result of this fraud is peculiar. Saffron is largely used for giving an agreeable color to medicinal preparation, so it is added to medicines in a prescription. This mineral matter is

apt to destroy the effect of other substances in the mixture, and may easily bring about changes in them that will result in poisoning the patient.

Let us now turn to the distinctively microscopical class of examinations and observe the facility of identification. Starch grains taken from different drugs, under the microscope are as conspicuously different as are larger objects. The same is true when they are modified in appearance by moist heat. The presence of such grains often shows that the drug has been partly exhausted of its activity. Powdered elecampane illustrates a very large class of drugs that do not contain any starch. If we find starch grains in any of these powders, we know that there must be an admixture. The various forms of crystals of calcium oxalate are very distinctive, the particular form being always the same in a given drug. Merely glancing at the powder under the microscope would identify a drug by this means. Ground olive pits have been used to the extent of hundreds of tons for adulterating such important drugs as ipecac, gentian, belladonna, and aconite. While stone cells occur in many drugs, similar to those of the olive pit, they are absent from most, and their characteristic appearance is sufficient for ready detection. The very similar stone cells from cocoa nut shells have been largely used to adulterate chocolate, but when compared with the powders of chocolate under the microscope they could not fail of detection.

Plant hairs are often so characteristic as to insure instant recognition. The stellate hairs of the chestnut leaf, one of the favorite articles used to adulterate medicinal leaves and herbs, are very distinctive; the peculiar hairs of stramonium and spurious henbane have already been mentioned. Genuine and spurious matico are easily distinguished, the latter having only about one-third the medicinal activity of the former. Its hairs are large, strong, and thick-walled, the cavity being little more than a faint line. The hair of the genuine, on the other hand, is nearly all cavity, its wall so thin that the hair frequently collapses.

It is earnestly hoped that this presentation of the subject may lead some here to interest themselves, at least a little, in this matter. The attention of this association has been chiefly directed to other things than drugs. Important as those subjects are, your aid is equally needed in the drug field. There are only a few of us to struggle with this great subject. Efforts to secure just action by the final authorities are met by the most energetic and often very plausible misrepresentations by interested parties, to the great detriment of the cause, and there is great need of your moral support in promoting public interest in the rigid enforcement of the laws regarding pure drugs.

THIRD DAY.

SATURDAY—MORNING SESSION.

Mr. J. P. Street introduced a resolution approving national legislation regulating the composition and sale of insecticides and fungicides. The matter was referred to the committee on resolutions. (See page 189.)

REPORT ON PHOSPHORIC ACID.

By J. M. McCANDLESS, *Referee*.

On May 19, 1908, the referee sent out a letter to twenty-one chemists, quoting the recommendations made by the association, as follows:

(1) That the referee on phosphoric acid take up for report at the next meeting of the association methods applicable under American conditions to the official examination of basic slag phosphates.

(2) That the subject of an accurate determination of iron oxid and alumina in rock phosphates be examined by the referee on phosphoric acid and an official method be recommended to the association next year.

(3) That a number of chemists be requested to send to the referee on phosphoric acid samples of the citrate ammonia solution employed by them, and that the referee examine such samples as to neutrality and that such examination be reported to the chemists at the next annual meeting.

In compliance with these instructions the referee requested those who desired to cooperate in the work to send him a bottle (200 cc) of their solution of ammonium citrate and a short statement of the method used in making the samples neutral.

In response to this letter the referee received nine samples of ammonium citrate solution for examination and forwarded to ten chemists three samples each, one of pulverized brown Tennessee rock, one of pulverized Florida rock, and one of a synthetic solution made from microcosmic salt, recrystallized potash-alum, ferrous ammonium sulphate, calcium carbonate, magnesium sulphate, and calcium fluorid, so that 100 cc would represent 1 gram of substance, and on that basis the solution should contain exactly 3 per cent of ferric oxid and 2 per cent of alumina.

A letter of instructions was forwarded with the samples requesting that the co-operators test the following methods for iron and alumina, it being deemed best to restrict the work to these phases:

METHODS FOR THE DETERMINATION OF IRON AND ALUMINA IN PHOSPHATE ROCK.

It is recommended that before beginning the work each analyst make up for himself a synthetic solution from C. P. chemicals, containing 10 grams of microcosmic salt, 10.4 grams of calcium carbonate, 0.050 gram of magnesium oxid or its equivalent in magnesium sulphate, 0.300 gram calcium fluorid. To these should be added accurately known weights of C. P. crystallized potash, or ammonia alum, and ferrous ammonium sulphate or iron wire. The material should be dissolved in hydrochloric acid and water and made up to a liter. The methods should be tried upon this solution to acquire confidence and applied to the referee's samples, using the following methods:

Gladding method.

Dissolve 4 grams of the rock in 30 cc dilute hydrochloric acid (1 to 1), heating just below the boiling point for half an hour. Filter into a 200 cc flask, add a few drops of nitric acid, and boil to oxidize the iron; cool and dilute to mark. Take 50 cc, containing 1 gram, and run into 20 cc of a solution of C. P. caustic potash, made by dissolving 500 grams of caustic potash free from alumina, in distilled water and diluting to one liter. Digest in water bath at 70° for one hour, stirring occasionally. Let the precipitate settle and filter on a large paper, first decanting the supernatant liquid on the paper and finally washing on the precipitate. Wash two or three times with hot water. To the filtrate add 1 gram of ammonium phosphate; acidify with hydrochloric acid. Add ammonia until a permanent precipitate is formed; add dilute hydrochloric acid, drop by drop, until it is just dissolved.

Add a mixture of 15 cc neutral ammonium acetate solution and 5 cc acetic acid (30 per cent) and digest for half an hour at 70° C., by which time the precipitation is complete. Filter, washing five or six times with hot ammonium acetate solution (10 per cent), stirring up the precipitate with the jet each time. Ignite with a low flame till the paper is charred, increase the heat until the paper is consumed, then blast for a minute.

The precipitate is $AlPO_4$, and its weight multiplied by 0.418 gives the Al_2O_3 . Gladding determines the iron oxid volumetrically by the bichromate method in a solution of the precipitate of iron oxid and calcium phosphate thrown down by the caustic potash, or by the same method in a separate solution of 5 grams of the rock in dilute hydrochloric acid (1 to 1).

Glaser method.

Boil 3 grams of phosphate rock thirty minutes in 30 cc concentrated hydrochloric acid. Make up to 300 cc and filter off 100 cc. Add 25 cc concentrated sulphuric acid; shake and allow to stand a few minutes; add 100 cc strong alcohol and cool. Make up to 250 cc with alcohol and allow to stand thirty minutes; filter off 100 cc or 0.4 gram and evaporate in a large beaker to expel alcohol.

Transfer to a small Griffin beaker, boil, remove from flame, and make slightly alkaline with ammonia. Boil to neutrality, cool, filter, and wash with boiling ammonium nitrate solution. Burn and weigh, weight divided by 2 = oxids of iron and aluminum.

Proposed modification of acetate method.

Weigh 2.5 grams of phosphate rock into a 250 cc flask; cover with 25 cc of concentrated hydrochloric acid; keep just below the boiling point for thirty minutes; dilute and cool; make up to the mark; filter off 50 cc, equivalent to one-half gram of rock; add a few drops of nitric acid, to oxidize any ferrous iron, and boil.

Add ammonia until the precipitate formed dissolves slowly on agitation. Then cool to about 15° C., neutralize, adding dilute ammonia drop by drop until the precipitation is complete. Clear up with dilute hydrochloric acid added drop by drop, slowly and with frequent shaking toward the last until the solution is clear. Make a solution of ammonium acetate by neutralizing strong ammonia with acetic acid sp. gr. 1.04; to 15 cc of this solution add 5 cc of acetic acid, sp. gr. 1.04, in a tall beaker having a capacity of about one liter; fill the beaker about seven-eighths full with hot water, so that the mixture will have a temperature of 70° to 75° C.; pour the solution of phosphate in a thin stream into the dilute hot solution of the ammonium acetate, stirring constantly. The precipitated phosphates of iron and aluminum are allowed to settle, and after becoming clear the greater part of the supernatant fluid is siphoned off, the beaker is filled up again with hot water at about 70°, again allowed to settle, and the supernatant fluid is siphoned off.

The remainder in the beaker is now filtered off on a large, rapid filtering paper (S. & S. black band ashless) washed thoroughly with hot water containing ammonium nitrate, keeping the precipitate on the filter well stirred up with a strong jet from the wash bottle. Ignite at a low temperature, till the paper is charred, increase heat until the paper is fully consumed, and finally blast for a minute. The weight of the precipitate in centigrams gives the percentage of the mixed oxids.

It is desired that in the last two methods the percentage of the mixed oxids of iron and alumina be given and also that the oxid of iron be determined separately by any volumetric method preferred by the analyst, always observing the precaution of oxidizing the organic matter to be found in solutions of phosphate rock by digesting with potassium chlorate and boiling off the excess of chlorine previous to the reduction and titration.

The referee desires to remind the analysts cooperating in this work that it has been undertaken under the auspices of the A. O. A. C. for the purpose of establishing, if possible, a standard method for the estimation of iron and alumina which should have the indorsement of the association. At present all is chaos, and when two chemists differ on iron and alumina it is impossible to say who is right and who is wrong.

The great majority of rock sales to-day are settled either by Gladding method or by the Glaser method, as outlined above.

The referee submits the above modification of the acetate method, which he believes to be simpler and fully as accurate as the others, and will welcome the comments and criticisms of the analysts when they have completed the work on the three samples by the methods outlined above. In regard to the synthetic solution sent, the referee would say that it was not practicable to send more than 300 cc to each analyst, but that in his opinion one-half gram, or 50 cc, is sufficient for any of the tests required, and used in this way there is sufficient of the synthetic solution for six tests.

Reports were received from six chemists cooperating in the work on iron and alumina, whose results are given in the following tables:

Determination of iron and alumina in Tennessee and Florida rock.

TENNESSEE ROCK.

Analyst.	Determination.	Method of—				
		Gladding.	Glaser.	McCandless.	Veitch.	Von Grueber.
		<i>Per cent.</i>	<i>Per cent</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Stillwell and Gladding, New York City.	Fe ₂ O ₃	2.79-2.79				
	Al ₂ O ₃	3.50-3.44				
	Total.....	6.29-6.23	6.80	6.20		
	Average.....	6.26		6.10		
F. B. Carpenter, by R. Henry, Richmond, Va.	Fe ₂ O ₃	3.17				
	Al ₂ O ₃	3.50				
	Total.....	6.67		6.48	7.20	
G. Farnham, Jarecki Chemical Co.	Fe ₂ O ₃	3.06	3.06			
	Al ₂ O ₃	2.29	3.92			
	Total.....	a 5.35	6.98			
P. Rudnick, by G. F. Beyer, Chicago, Ill. ^b	Fe ₂ O ₃	3.22				3.22
	Al ₂ O ₃	3.21				3.68
	Total.....	6.43	7.04	6.19		6.90
	Average.....					
McCandless, Burton, and Atkinson, Atlanta, Ga.	Fe ₂ O ₃	3.04				
	Al ₂ O ₃	3.26				
	Total.....	6.30	6.48	6.22		
S. H. Wilson, Georgia Department of Agriculture.	Fe ₂ O ₃	2.91				
	Al ₂ O ₃	3.21				
	Total.....	6.12	5.86	5.90		
O. M. Shedd, Kentucky station.	Fe ₂ O ₃		2.92-2.92			
	Al ₂ O ₃					
	Total.....		6.39-6.49	4.32-4.13		
	Average.....		6.44	a 4.22		
Average.....		6.35	6.60	6.19		

^a Omitted from average.

^b Per cent calculated from average of three determinations.

Determination of iron and alumina in Tennessee and Florida rock—Continued.

FLORIDA ROCK.

Analyst.	Determination.	Method of—				
		Gladding.	Glaser.	McCandless.	Veitch.	Von Grueber.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Stillwell and Gladding, New York City.	Fe ₂ O ₃	1.71		1.70-1.75		
	Al ₂ O ₃	1.49		.92-1.00		
	Total.....	3.20	3.51	2.62-2.75		
	Average.....			2.68		
F. B. Carpenter, by R. Henry Richmond, Va.	Fe ₂ O ₃	1.85				
	Al ₂ O ₃	.94				
	Total.....	2.79		2.73	2.89	
G. Farnham, Jarecki Chemical Co.	Fe ₂ O ₃	1.65	1.65			
	Al ₂ O ₃	.66	1.33			
	Total.....	2.31	2.98			
P. Rudnick, by G. F. Beyer, Chicago, Ill. ^a	Fe ₂ O ₃	1.86				1.86
	Al ₂ O ₃	1.03				1.53
	Total.....	2.89	3.48	1.95-1.97		3.43
	Average.....			1.96		
McCandless, Burton, and Atkinson, Atlanta, Ga.	Fe ₂ O ₃	1.73				
	Al ₂ O ₃	.94				
	Total.....	2.67	3.90	2.72		
S. H. Wilson, Georgia Department of Agriculture.	Fe ₂ O ₃	1.68				
	Al ₂ O ₃	.90				
	Total.....	2.58	2.96	2.62		
O. M. Shedd, Kentucky station.	Fe ₂ O ₃		1.68-1.66			
	Al ₂ O ₃		1.67			
	Total.....		2.96-2.98	1.50-1.69		
	Average.....		2.97			
Average.....		2.74	3.30	2.69		

^a Per cent calculated from average of three determinations.

Determination of iron and alumina in synthetic solution.[Synthetic solution—made to contain 3 per cent Fe_2O_3 and 2 per cent Al_2O_3 , or 5 per cent combined oxids.]

Analyst.	Determination.	Method of—				
		Gladding.	Glaser.	McCandless.	Veitch.	Von Grueber.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Stillwell and Gladding, New York City.	Fe_2O_3	2.60		2.50		
	Al_2O_3	3.68		2.73		
	Total.....	^a 6.28	^a 6.12	5.23		
F. B. Carpenter, by R. Henry, Richmond, Va.	Fe_2O_3					
	Al_2O_3					
	Total.....			5.01	5.68	
G. Farnham, Jarecki Chemical Co.	Fe_2O_3	3.08	3.08			
	Al_2O_3	1.86	1.86			
	Total.....	4.94	4.94			
P. Rudnick, by G. F. Beyer, Chicago, Ill. ^b	Fe_2O_3	3.11		3.11		
	Al_2O_3	2.15		1.50		
	Total.....	5.26	5.62	4.61		
McCandless, Burton, and Atkinson, Atlanta, Ga.	Fe_2O_3	3.05				
	Al_2O_3	2.06				
	Total.....	5.11	5.55-5.60 (5.57)	5.07-5.95 (5.06)		
S. H. Wilson, Georgia Department of Agriculture.	Fe_2O_3	2.91				
	Al_2O_3	2.55				
	Total.....	5.46	5.40	5.18		
O. M. Shedd, Kentucky station.	Fe_2O_3		3.12-3.12 (3.12)			
	Al_2O_3					
	Total.....		4.92-4.98 (4.95)	4.10-4.38 ^a (4.24)		
Average.....		5.28	5.29	5.02		

^a Omitted from average.^b Per cent calculated.

The determination of iron and alumina by various modifications (Shedd).^a

GLADDING METHOD.

Modification.	Florida rock (25198).	Tennessee rock (25199).	Referee's synthetic solution (25200), 50 cc.	Shedd's synthetic solution, 50 cc.
Al_2O_3 by subtracting FePO_4 from weight of $\text{FePO}_4 + \text{AlPO}_4$ and multiplying by 0.419:	Per cent.	Per cent.	Per cent.	Per cent.
First determination.....				0.0108
Second determination.....				.0111
Mean.....				.0110
Theory.....				.0100
Fe_2O_3 obtained from the precipitate of $\text{FePO}_4 + \text{AlPO}_4$ obtained above:				
First determination.....				.0193
Second determination.....				.0195
Mean.....				.0194
Fe_2O_3 obtained from independent portions of 50 cc of the solution:				
First determination.....				.0194
Second determination.....				.0194
Mean.....				.0194
Theory.....				.0194

MODIFIED ACETATE METHOD.

Al_2O_3 by subtracting FePO_4 , etc., as above:				
First determination.....	-0.10	1.25	0.0051	-0.0020
Second determination.....	-.05	1.09	.0058	
Mean.....	-.025	1.17	.0055	
$\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$ by halving the weight of phosphates:				
First determination.....	1.50	4.32	.0205	.0158
Second determination.....	1.69	4.13	.0219	
Mean.....	1.60	4.23	.0212	
Fe_2O_3 from independent 50 cc portions of the solution:				
First determination.....	1.74	3.03	.0157	
Second determination.....	1.69	2.94	.0157	
Mean.....	1.72	2.99	.0157	

GLASER METHOD.

Al_2O_3 by subtracting FePO_4 , as above:				
First determination.....	1.16	3.05	0.0082	0.0047
Second determination.....	1.17	3.13	.0084	
Mean.....	1.17	3.09	.0083	
$\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$ by halving the weight of phosphates:				
First determination.....	2.96	6.39	.0246	.0239
Second determination.....	2.98	6.49	.0249	
Mean.....	2.97	6.44	.0248	
Fe_2O_3 from independent 50 cc portions of the solution:				
First determination.....	1.68	2.92		
Second determination.....	1.66	2.92		
Mean.....	1.67	2.92		

^a Analyses made by O. M. Shedd, of the Kentucky station, but received too late to incorporate in the report.

DISCUSSION OF RESULTS.

P. Rudnick (results by G. F. Beyer): Commenting on the results in general, I do not believe that any of the methods proposed are preferable to the modified Von Grueber method in point of simplicity, rapidity, accuracy, and general applicability to various kinds of rock. Although the results obtained for ferric oxid in the synthetic solution prepared in this laboratory are high, they agree very well with the results obtained by the determination of iron in the precipitate from the method proposed by you, and the results on aluminum by the modified Von Grueber method are certainly very close to the calculated quantity. Although I have not had time nor opportunity to prove the point, I am inclined to believe that ammonium acetate is not sufficient to prevent the partial hydrolysis of the aluminum phosphate, and that ammonium nitrate is more efficient in this respect. I believe the fairly good agreement between the results by the Glaser method and the modified Von Grueber method obtained in this work, as well as at other times, supports this view.

S. H. Wilson: For simplicity and ease of execution the McCandless laboratory method leaves little to be desired.

Remarks by the referee: On the whole the results seem to be encouraging and to show that all three of the methods for which instructions were sent are capable of giving good results. One analyst used the Von Grueber method, another the Veitch method. A study of the results on the synthetic solution, in which the percentages of iron and alumina are accurately known, reveals a tendency on the part of those getting the lowest results on the phosphate rocks to get them also on the solution and vice versa; excluding the lowest and highest results, the agreements and approximations to the truth are about as good as would be found in the determination of other elements, as, for instance, phosphoric acid by the accepted methods.

The referee would call attention to the fact that this subject has been taken up by the National Fertilizer Association, and would recommend cooperation between the next referee and the committee of that association, with a view to reaching a decision as to what method shall be adopted.

EFFECT OF DILUTE AND CONCENTRATED HYDROCHLORIC ACID ON PYRITES IN PHOSPHATE ROCK.

The referee also requested the analysts cooperating to test the effect of dilute (1 to 1), and concentrated hydrochloric acid as to its solvent effect on pyrites, present to a greater or less extent in nearly all phosphate rock.

It has been claimed on the one hand that dilute hydrochloric acid (1 to 1) fails to dissolve all the iron and alumina, especially when the latter is present in the form of clay; it has been claimed, on the other hand, that concentrated hydrochloric acid, while it dissolves the alumina and iron oxides better than the dilute, also decomposes pyrites present in the phosphate rock and therefore yields too high a percentage of iron. It is desired that the analysts test this latter point as follows:

Procure a sample of freshly pulverized pyrites and weigh half a gram into a 250 cc flask, cover with 25 cc of hydrochloric acid (1 to 1), heat just below boiling for thirty minutes, dilute with 100 cc of water, shake, allow to settle, decant the liquid, repeat the washing by two or more treatments with 125 cc of cold water slightly acidulated with hydrochloric acid, followed by decantation. This preliminary treatment is to remove any oxid or sulphate of iron already existing in the pyrites. Have ready 2.5 grams of phosphate rock, add it to the flask on top of the washed pyrites, then cover with 30 cc of concentrated hydrochloric acid, heat just below boiling for thirty minutes, cool and make up to the mark. Determine the iron volumetrically in an aliquot of the solution and compare the results with that obtained from a similar treatment of phosphate rock and pyrites with dilute hydrochloric acid (1 to 1) the second time.

Only two chemists beside the referee took part in this work.

Solvent effect of dilute and concentrated hydrochloric acid on pyrites.

Analyst.	Phosphate rock.	2.5 grams rock and 0.5 gram pyrites.	
		Concentrated HCl.	Dilute HCl.
G. F. Beyer, Chicago, Ill.....	3.51-3.51	3.5-3.60	3.60-3.51
G. Farnham, Jarecki Chemical Co.....		2.91	3.74
J. M. McCandless, Atlanta, Ga.....	3.48-3.50	3.49-3.52	3.50-3.47

While one of the results in the above table must be explained, the referee is convinced from a number of tests made years since that neither dilute nor concentrated hydrochloric acid has any appreciable effect on pyrites, and would therefore recommend the use of the concentrated acid in the solution of phosphate rock, heating for a definite time.

Of course, the use of sulphuric acid of 50° B. (which also has no action on pyrites), followed by solution in dilute hydrochloric acid, would more nearly approximate actual conditions, and it might be well if the next referee would investigate this method of solution as compared with simple solution in concentrated hydrochloric acid.

EXAMINATIONS OF SOLUTIONS OF AMMONIUM CITRATE FOR NEUTRALITY.

As the referee was to decide whether the solutions were neutral or not, and as no two chemists agree on the exact point of neutrality, whether from lack of sensitiveness of the indicators, or color-blindness on the part of the operators, he decided to make an analysis of each sample according to the method outlined in his last report to the association, and be guided by those results in deciding upon neutrality. The following method of analysis was adopted:

Twenty-five cubic centimeters of each solution was pipetted into a 250 cc flask, made to mark, shaken, and 25 cc pipetted into a distillation flask; to the solution in the flask, 40 cc of fourth-normal caustic soda solution were added, and the contents of the flask distilled into 20 cc of half-normal acid, continuing the distillation until the volume of the distillate measured from 65 to 70 cc. The ammonia in the distillate was then titrated by means of tenth-normal alkali. The residue in the retort was washed into an Erlenmeyer flask, excess of standard acid added, then a few drops of phenolphthalein, and the excess estimated by means of tenth-normal alkali. From the result the weight of citric acid originally combined with the ammonia was calculated. Calculating from the formula of the pure salt, $(\text{NH}_4)_2\text{C}_6\text{H}_5\text{O}_7$, that the ratio of ammonia (NH_3) to citric acid was as 1 to 3.765, a basis of comparison was established. The results obtained are given in the table below. As only three official chemists sent their solutions, the analysts are designated by number and not by name.

Determination of neutrality of ammonium citrate solutions.

Number of analyst.	Ammonia in 25 cc of diluted solution=24 cc original.	Citric acid in 25 cc of diluted solution=24 cc original.	Ratio of ammonia to citric acid.	Ratio in neutral salt $(\text{NH}_4)_2\text{C}_6\text{H}_5\text{O}_7$.	Reaction with corallin.
	<i>Milligrams.</i>	<i>Milligrams.</i>			
1.....	113.9	433.2	1:3.903	1:3.765	Neutral.
2.....	109.3	412.8	1:3.775	1:3.765	Alkaline.
3.....	104.9	424.96	1:4.051	1:3.765	Acid.
4.....	113.7	433.9	1:3.816	1:3.765	Neutral.
5.....	110.5	430.08	1:3.891	1:3.765	Slightly acid.
6.....	111.5	436.48	1:3.915	1:3.765	Acid.
7.....	108.7	421.1	1:3.874	1:3.765	Slightly acid.
8.....	104.7	398.7	1:3.808	1:3.765	Neutral.
9.....	102.8	430.7	1:4.189	1:3.765	Acid.

In this table all the solutions which showed materially more citric acid than 3.765 parts to 1 of ammonia also showed a decidedly acid reaction to corallin.

It appears that some chemists prepare their ammonium citrate solution by treating the citric acid with excess of ammonia and then leave the hot solution to neutralize itself, or finally adjust it, some by means of red and blue litmus paper, others by corallin. Some state that they have never been successful in the use of corallin; others adjust finally by means of the alcoholic solution of calcium chlorid. In the opinion of the referee, if a chemist has succeeded in getting his solution neutral or practically so, he will almost certainly put it out of joint by attempting to make it exact with the calcium chlorid solution. The referee finds that an alcoholic calcium chlorid solution which is exactly neutral to corallin is acid to phenolphthalein, and alkaline to cochineal; that after the precipitation of the citric acid from 10 cc of the ammonium citrate solution by 50 cc of the calcium chlorid solution, calcium citrate still remains in solution in the filtrate, which may be proved by boiling some of the clear solution, when a precipitate of calcium citrate will appear. The presence of this salt, in the opinion of the referee, renders the use of cochineal as indicator unreliable. One of the solutions in the above table, which is the most acid of all by analysis, was neutralized in this way. There are materials (notably fertilizers containing bone) on which a slight difference in neutrality of the ammonium citrate solution makes a great difference in the results. It is a reproach to the association that it has suffered the matter to remain in its present condition so long. While the referee has a strong personal conviction that the only proper method of making the solution neutral is by analysis and calculation of the exact quantity of ammonia or citric acid to be added to it, still he hesitates to urge it officially, as no work has yet been done by any other referee along this line, and because the referee is himself no longer an official chemist.

The referee desires to acknowledge the valuable aid and suggestions of Mr. J. Q. Burton in all of this work and the analytical assistance of Mr. F. C. Atkinson.

THOMAS SLAG.

By J. B. LINDSEY.

Thomas slag or basic phosphatic slag is a by-product in the modern method of steel manufacture from ores containing noticeable quantities of phosphorus. The process of removing the phosphorus from the ore was discovered by the English engineers Gilchrist and Thomas and, briefly stated, consists in adding to the so-called "converter" containing the molten ore a definite quantity of freshly burned lime, which after a powerful reaction is found to be united with the phosphorus and swims upon the surface of the molten steel in the form of a slag.

COMPOSITION.

The composition of the Thomas or Belgian slag varies according to the character of the ore and the success of the process for removing the impurities. The following figures show such variations: ^a

	Per cent.
Phosphoric acid.....	11-23
Silicic acid.....	3-13
Calcium oxid (lime).....	38-59
Ferrous and ferric oxids.....	6-25
Protoxid of manganese.....	1- 6
Alumina.....	0.2- 3.7
Magnesia.....	2- 8
Sulphur.....	0.2- 1.4

^a Adolf Mayer, *Agricultural Chemie*, 6th ed., vol. 2, pt. 2, pp. 138-139.

More or less metallic iron is inclosed in the coarse slag which is generally thoroughly removed from the ground material by the magnet.

MANURIAL VALUE.

The manurial value of the slag was not recognized for a long time. Finally experiments revealed that a considerable portion of its phosphoric acid was soluble in dilute citric and carbonic acids, which led to successful field experiments. The only preparation of the slag for fertilizing purposes, when its value was first recognized, consisted in having it finely ground in especially prepared mills, so that 75 per cent would pass through a sieve with perforations 0.17 mm in diameter. This requirement was suggested by M. Fleischer, who used the slag with much success in improving the condition of marsh and meadow lands.

METHODS FOR DETERMINING AVAILABILITY AND ADULTERATION.

Previous to 1890, by means of pot experiments, as well as by laboratory investigations, Wagner demonstrated that the phosphoric acid in different slags of the same degree of fineness varied in its availability from 30 to 90 or more per cent, and, further, that many brands were adulterated with Belgian or other insoluble mineral phosphates. The method therefore of determining the value of a slag by the percentage of total phosphoric acid present and the degree of fineness was of secondary importance.

In order to detect adulteration with mineral phosphates, Wagner originally used a dilute solution of citrate of ammonia and free citric acid.^a The phosphoric acid in all of the mineral phosphates was sparingly soluble in such a reagent, while an unadulterated, high-grade slag gave up 80 to 90 parts of its phosphoric acid. Further investigations on various soils with many brands of slag made clear that the results obtained from pot experiments corresponded quite well with those secured by means of the citric-acid solution. This may be illustrated as follows:

Determination of the availability of phosphoric acid.

Brand of slag.	Phosphoric acid available.	
	In citric-acid solution.	In pot experiments.
1	100	100
2	85	80
3	81	72
4	72	72
5	73	66
6	76	63
7	39	40
8	48	38
9	42	38
10	45	31
11	38	30

Results similar to these were secured by Maercker,^b who stated that "the results removed all doubt that the citrate solubility and plant experiments were so nearly proportioned that one had the same right to value the slag according to its content of phosphoric acid soluble in citrate solution as to value the superphosphate by its content of water-soluble phosphoric acid."

^a Chemiker Zeitung, 1895, No. 63; also Düngungsfragen, 1896, No. 1, p. 16.

^b Landw. Presse, 1895, No. 82.

As a result of these investigations the union of German experiment stations at its meeting at Kiel, in September, 1896, adopted the method ^a of determining the relative value of the slag according to its phosphoric acid solubility in a 2 per cent citric acid solution and did away with the previous standard of total phosphoric acid and fineness.

Wagner, as well as Maercker, repeatedly called attention to the fact that experiments both in the laboratory and with plants gave positive evidence that those slags of like phosphoric acid content which were richest in silicic acid gave the best results. G. Hoyeremann, working independently, came to similar conclusions. At the present time, according to Wagner, practically all of the iron works treat the molten slag as it flows from the converter with hot quartz sand, with the result that the availability of the phosphoric acid is increased from 10 to 30 per cent.^b

CHEMICAL COMBINATION OF PHOSPHORIC ACID IN SLAG.

The form in which the phosphoric acid exists in the slag has never been fully explained. It was formerly supposed that it was combined with lime as a tetracalcium phosphate and the latter being less stable than tricalcium phosphate became easily available to the plants by being decomposed, under the influence of dilute acids, into the calcium salt of the dissolving acid and dicalcium phosphate. The tetralime phosphate, however, has never been made artificially,^c although it has been recognized by the aid of the microscope in the slag and exists as a mineral under the name of isoklas.

More recent investigations having shown, as already indicated, that those slags richest in silicic acid of like phosphoric acid content gave the best results, the conclusion followed that a part of the lime must be in the form of lime silicate. It is now generally held, especially by Wagner,^d that the phosphoric acid is combined in the slag as a double salt of tricalcium phosphate and calcium silicate and that in this form the roots are able to utilize it. It is also believed probable that some of the phosphoric acid is more or less united with iron as a basic iron phosphate.

THE USE OF PHOSPHATIC SLAG.

Basic slag has been shown to work especially well upon sour marsh and meadow lands, upon porous, well-aired soils rich in humus, and upon sandy soils deficient in lime.

When a rapid development of the crop is not desired, the slag may be used exclusively in place of acid phosphate. On the other hand, in cases when it is feared that the crop will not mature early enough upon heavy, cold land and in high altitudes where the season is short, acid phosphate should be given the preference.

The phosphoric acid in slag is comparable in its quickness of action to nitrogen in barnyard manure, tankage, and green crops; and the phosphoric acid in acid phosphate to the action of nitrogen in nitrate of soda. A combination of slag and sulphate of potash (500 pounds of slag and 100 pounds of potash) has been found to work especially well upon grass land and to be very favorable to the development of clover.

^a Method slightly modified from the original. Present method described in König's *Untersuchung landwirtschaftlich und gewerblich wichtiger Stoffe*, 3d ed., pp. 173-174.

^b Loc. cit.; also Wagner, *Anwendung künstlicher Düngemittel*, pp. 74-75.

^c Hilgenstock, *Jahresber. chem. Technologie*, 1887, p. 282, after Adolf Mayer, loc. cit.

^d Loc. cit.

QUANTITY OF SLAG PER ACRE.

If the soil is particularly deficient in phosphoric acid, one can use as high as from 800 to 900 pounds of slag to the acre, plowed in and supplemented with 200 pounds of acid phosphate in the hill or drill.

If, on the contrary, the soil is naturally rich in phosphoric acid or has been made so by large additions of slag for several consecutive years (1,000 or more pounds yearly) then it is necessary only to replace from year to year the amount removed by the crop. In such cases Maercker states that one part of phosphoric acid in basic slag is as valuable as an equal amount in acid phosphate.

VALUATION OF PHOSPHORIC ACID IN BASIC SLAG.

By H. D. HASKINS.

Thomas basic slag is being used in Massachusetts and some of the other New England States more extensively each year, and it is therefore highly desirable that more satisfactory methods of analyzing and valuing this product be worked out by the association.

At the last meeting of the association the referee on phosphoric acid recommended that the phosphoric acid in Thomas basic slag be valued by the degree of fineness of the slag along the same lines as are employed in the case of ground bone, but the recommendation did not specify the diameter of the openings in the sieve used in the mechanical separation. If valued by its fineness the diameter of the openings in the sieve used for this purpose is obviously of great importance, as is shown by the following results: Two samples of slag were analyzed mechanically by the use of 100-, 50-, and 25-mesh sieves, the latter having circular openings one-fiftieth of an inch in diameter.

Comparison of amount and value of phosphoric acid available by using sieves of different sized mesh.

Sample.	Total P_2O_5 .	Mechanical analysis.						Valuation.		
		100-mesh sieve.		50-mesh sieve.		25-mesh sieve.		100-mesh sieve.	50-mesh sieve.	25-mesh sieve.
		Fine.	Coarse.	Fine.	Coarse.	Fine.	Coarse.			
	<i>Per cent.</i>	<i>Per ct.</i>	<i>Per cent.</i>	<i>Per ct.</i>	<i>Per cent.</i>	<i>Per ct.</i>	<i>Per cent.</i>	<i>Dollars.</i>	<i>Dollars.</i>	<i>Dollars.</i>
1	17.45	68.47	31.53	89.64	10.36	93.72	6.28	12.86	13.59	13.74
2	17.96	71.83	28.17	90.74	9.26	94.97	5.03	13.36	14.04	14.18

An average of the two samples shows that 94.34 per cent of the slag passes a 25-mesh sieve, 90.19 per cent passes a 50-mesh sieve, and only 70.15 per cent passes a 100-mesh sieve. Looking at the matter from the point of valuation, we find that allowing 4 cents for the phosphoric acid in the fine and 3 cents for the phosphoric acid in the coarse slag, the average valuation of the two samples of slag by use of the 25-mesh sieve would be \$13.96, by use of the 50-mesh sieve \$13.81, and by use of the 100-mesh sieve only \$13.11; a difference of nearly \$1 per ton in the extremes.

The next question to be considered is whether this method of valuing the phosphoric acid in basic slag is a safe one for us to adopt. During the month of August the writer's attention was called to a product put forth in large quantities in northern New York. The material was a waste product said to be largely apatite and quartz, coming from iron ore used in that locality. It was finely ground, 69.44 per cent passing a 100-mesh sieve, and contained 16.78 per cent of total phosphoric acid; the phos-

phoric acid in this material, however, by the Wagner method of analysis showed only 1.029 per cent of available phosphoric acid or 6.13 per cent of the whole, while the basic slag by this same method showed 15.48 per cent of available phosphoric acid or 87.4 per cent of the whole. The point in making mention of this apatite is to show that in case the phosphatic slag is adulterated with material of this nature the mechanical method of valuing the slag would prove decidedly misleading, and it is because of this that the method of valuing the phosphoric acid in basic slag has become obsolete in European countries.

During the past year the writer has had experience with the Wagner method of determining available phosphoric acid in basic slag and the valuations of this material that appear in our fertilizer bulletin have been based on this method.

The Wagner method as used in foreign countries has shown results agreeing closely with those obtained in field trials. It is as follows:

Weigh 5 grams of the slag and transfer it to a half-liter, bottle-shaped flask containing 5 cc of alcohol to prevent the slag from adhering to the flask. Make up to the mark with a 2 per cent citric acid solution at 17.5° C. The flask is fitted with a rubber stopper and put at once into a rotary apparatus for thirty minutes, making thirty to forty revolutions per minute. At the end of a half hour the solution is immediately filtered and the phosphoric acid is determined in an aliquot part of the clear solution by means of molybdic solution in the usual manner.

The analysis of two samples of slag by this method at the Massachusetts experiment station shows the following close agreement. No. 1, available phosphoric acid, 15.42 and 15.38; No. 2, 15.81 and 15.75.

In case of a bona fide sample of basic slag the valuation based upon mechanical analysis by use of a 100-mesh sieve agrees closely with the valuation based on the availability of the phosphoric acid by the Wagner method. In case, however, of a sample of slag adulterated with the natural mineral phosphate, the valuation based on mechanical fineness is obviously open to severe criticism. I think this question of sufficient importance to warrant a motion that I would herewith make, that the referee on phosphoric acid be instructed to make a study of the Wagner method of analysis with samples of basic slag and natural mineral phosphates, with a view to its adoption as an official method for the determination of available phosphoric acid in basic slag.

The papers by Mr. Lindsey and Mr. Haskins relating to the valuation of phosphoric acid in basic slag were referred to Committee A for action on recommendations contained therein.

REPORT ON DAIRY PRODUCTS.

J. M. BARTLETT, *Referee.*

According to instructions given by vote of the association last year the referee has continued the study of analytical methods for condensed milks. The results reported at the last meeting indicated that the analysis of the sweetened product presented much greater difficulties than the unsweetened, particularly in the determination of fat; therefore, the referee decided to confine the work to one brand only, the sweetened milk. Twenty-six analysts signified a desire to cooperate, but not all of them were official chemists, many being commercial chemists more or less directly interested in food analysis.

SAMPLES OF MILK.

On about April 1, a can of sweetened condensed milk together with a copy of instructions was sent to each chemist requesting the same. It was first intended to get a quantity of milk in bulk, thoroughly mix it in the laboratory, and send out the samples

in bottles to insure uniformity, but a letter from the Borden Company, who furnished the samples, describing their process, whereby the milk is continuously agitated until it reaches the cans, convinced the writer that all cans from the same batch must be as uniform as it is possible to make them.

INSTRUCTIONS FOR ANALYSIS OF CONDENSED MILK.

Preparation of sample.

Mix thoroughly by transferring the contents of a can to some dish sufficiently large to thoroughly stir and make the whole homogeneous. Weigh 40 grams into a 100 cc flask and make up to the mark with water.

Total solids.

Method A.—Dilute a measured portion of the above 40 per cent solution with an equal amount of water. Use 5 cc of the diluted mixture and proceed as in the case of milk analysis according to the method given in Bulletin 107, page 117, Method I, drying either on sand or asbestos fiber.

Method B.—Use Leach's method which is as follows: Dilute a portion of the 40 per cent solution with an equal amount of water and take enough of this solution to represent 1 gram of the condensed milk. Put in a tared platinum dish which will hold at least 25 cc and still further dilute with water until the dish is nearly full, rinsing the pipette into the dish, then allow the dish to remain in contact with live steam for at least two hours after the last traces of the water have apparently been evaporated, then transfer to the drying oven for a few minutes, cool in the desiccator and weigh.

Ash.

Ignite the residue from total solids, cool and weigh in the usual manner.

Protein.

Determine nitrogen by Kjeldahl or Gunning method in 5 cc of the 40 per cent solution and multiply by 6.25.

Lactose.

Dilute 50 cc of the 40 per cent solution in a 250 cc flask to about 200 cc, add 6 cc of Fehling's copper sulphate solution, and make up to the mark, filter through a dry filter and determine lactose as follows:

Take 25 cc each of the copper sulphate and alkaline tartrate solution, add 50 cc water and bring to boiling, then add 25 cc of the filtered milk solution and boil two minutes (by longer boiling the sucrose appears to throw down some copper), remove from the lamp and allow to settle one or two minutes then filter on a gooch crucible in the usual manner, weighing the cuprous oxid after drying at 100 degrees. Give weights of cuprous oxid found as well as percentages of lactose so methods of calculation can be compared. Also, if possible, determine lactose by polariscope in this solution.

Sucrose.

Place 25 cc of the above solution, used to determine lactose, in a 100 cc flask. Add 50 cc of water and 0.75 of a gram of citric acid and heat on the steam bath for thirty minutes, nearly neutralize with sodium hydroxid and determine total sugars in 25 cc with Fehling's solution in the usual manner, giving the weight of cuprous oxid as well as the percentages of sucrose. Also determine sucrose by difference, subtracting the lactose, protein, fat and ash from total solids.

Fat.

Method A.—Determine by double extraction method. (See Bureau of Chemistry, Circular 32, p. 6.)

Method B.—By the Babcock centrifugal method using the modification given in Bulletin 107, page 123.

Method C.—The Gottlieb method, the directions for which are as follows:

Ten cubic centimeters of milk are measured into a glass cylinder three-fourths inch in diameter and about 14 inches long (see Landw. Vers. Sta., 40:6; a 100 cc burette or a eudiometer tube will do); 1 cc of concentrated ammonia is added and mixed well with the milk. The following chemicals are next added, in the order given: 10 cc of 92 per cent alcohol, 25 cc of washed ether, and 25 cc petroleum ether (boiling point

below 80° C.), the cylinder being closed with a moistened cork stopper and the contents shaken several times after the addition of each. The cylinder is then left standing for six hours or more. The clear fat solution is next pipetted off into a small weighed flask by means of a siphon drawn to a fine point (fig. 6, loc. cit.), which is lowered into the fat solution to within 0.5 cm of the turbid bottom layer. After evaporating the ether solution in a hood, the flasks are dried in a steam oven for two or three hours and weighed. This method is applicable to new milk, skim milk, buttermilk, whey, cream, cheese, condensed milk, and milk powder, but has been found of special value for determining fat in skim milk, buttermilk, cheese, and condensed milk. In the case of products high in fat, a second treatment with 10 cc each of ether and petroleum ether is advisable in order to recover the last trace of fat.

Chemists are requested to make at least two determinations by the methods given. On account of the quite large variations in the results reported by the chemists last year, the referee is very anxious to determine whether the differences are due to the inaccuracy of the methods or to the manner in which they are handled by different men. Everyone who has had much experience in making sugar determinations realizes how easy it is to get quite large variations in results by varying the method slightly.

If methods materially different from those given above are being used by anyone taking part in the work for the determination of sugars or fat in condensed milks, the referee will be glad to have results by such methods reported.

J. M. BARTLETT,
Referee.

L. G. MICHAELS,
Associate referee.

Eleven different chemists reported on the samples sent them and their results are given in the following table, together with some obtained at the Maine station:

Cooperative work on samples of sweetened condensed milk (percentage results).

Analyst.	Solids.			Protein N $\times 6.25$.	Lactose.	Sucrose.			Fat.	
	Dried on sand or asbestos.	Leach's method.	Ash.			Direct.	By difference.	Polariscope.	Double extraction.	Modified Babcock.
W. A. Brennon, Wisconsin station.....	72.44 73.14 73.06	72.08 73.19 72.90	1.81 1.82 1.66		11.47 11.56 11.56	44.76 44.50			8.10 8.24	8.32 8.10
Average.....	72.89	72.72	1.76	7.42	11.53	44.68	44.01	44.51	8.17	8.21
E. M. Bailey, Connecticut station.....	72.25 72.06	73.71 73.50							8.00 7.80	8.25 8.40
Average.....	72.15	73.60							7.90	8.33
J. M. Bartlett, Maine station.....	70.70 70.50				11.3 11.1					8.50 8.70
Average.....	70.60		1.70	7.20	11.2		43.24		8.66	8.60
Sidney Davis, department of health, New York City.....		73.13	1.72	6.96	11.72			44.09	9.00	
L. W. Fetzer, Maryland station.....	72.01 72.03	74.47 74.70	1.53 1.52	7.20 7.23	11.42 11.42				8.66 8.63	7.80 7.95
Average.....	72.02	74.53	1.53	7.22	11.42	37.85	44.20		8.65	7.90
H. B. Holland, Massachusetts station: Average of 4 tests.....	71.52		1.66	7.42					8.29	
Second sample, average of 3 tests.....	71.41								8.85	
C. H. Jones, Vermont station: Average of 2 tests.....	69.75	70.58	1.68	7.09	11.63	43.77	41.19		8.16	8.10
Second sample, average of 2 tests.....	72.20								8.56	8.55
C. P. Mout, Vermont board of health.....	70.00	72.51	1.51						6.6	

° Omitted from average.

° Dried on sand and extracted once.

° Extracted twice.

Cooperative work on samples of sweetened condensed milk (percentage results)—Cont'd.

Analyst.	Solids.		Ash.	Protein N×6.25.	Lactose.	Sucrose.			Fat.		
	Dried on sand or asbestos.	Leach's method.				Direct.	By difference.	Polariscope.	Double extraction.	Modified Babcock.	Gottlieb.
C. B. Morrison, Connecticut station (New Haven).....	68.75 68.97	70.55 70.81	1.95 2.05	7.06 7.06					8.19 8.04	7.2 7.5	 7.98
Average.....	68.86	70.68	2.00	7.06					8.12	7.35	
A. J. Patten, Michigan station	72.35 72.37	73.87 73.88	1.80 1.82	7.06 7.06	12.05 11.90					7.20 7.20	7.96 7.80
Average.....	72.36	73.88	1.81	7.06	11.95		44.63	43.65	7.01	7.20	7.88
Nelson & Landers, Binghamton, N. Y.	72.45		2.47	7.87	10.30	43.70	44.59		8.52	8.40	8.39
A. C. Whittier, Maine station.	70.70 70.80	73.20 73.33	1.73 1.72		11.02 11.06				8.54 8.45	8.70 8.70	7.89 7.78
Average.....	70.75	73.27	1.71	7.19	11.04		44.79	44.79	8.50	8.70	7.83
A. D. Meeds, Minneapolis, Minn.....	72.67 72.80	73.20 73.00	1.74 1.74	6.50 6.50	9.78 9.74	43.07 43.06	46.64 46.74		8.04 8.05	7.20 7.20	8.25 8.11
Average.....	72.74	73.10	1.74	6.50	9.76	43.07	46.68		8.05	7.20	8.18
J. C. Colcord, Maine station..	70.80								8.57		
Average.....	71.57	72.80	1.69	7.25	11.35	43.80	44.12	44.18	8.17	7.87	8.16

* Omitted from average.

COMMENTS OF ANALYSTS.

W. A. Brennon, Wisconsin station: The solids were determined in Method A by drying on sand five and a half hours and in B by drying three hours on steam bath and two hours in hot-air oven.

E. M. Bailey, Connecticut station: Total solids were determined in Method A by drying on sand in an 8 cm aluminum dish, there being enough sand to make a total of 50 grams weight. Evaporation was first made on live steam, with frequent stirring. The dish was then wiped dry and put in a drying oven at 100° for thirty minutes, cooled and weighed, then again heated three hours, and for seven hours.

Per cent.

Average for thirty minutes..... 72.74

Average for three hours..... 72.40

Average for seven hours..... 72.11

In Method B the manipulation was the same as in A, except that no absorbent was used. Average for heating thirty minutes was 73.58 per cent; for two hours, 73.60 per cent.

In the double-extraction method for fat the first and second extractions were made for eight hours each and the third for four hours. In the centrifugal method the pipette was rinsed into the bottle.

Lewis W. Fetzer, Maryland station: The methods involving dilution of a 40 per cent solution are very prone to cause an error, as it is next to impossible to dilute with a pipette or cylinder. Where this was directed I made a separate 20 per cent solution. The per cent of sucrose is better determined by difference, as the galactose which is formed by inverting the milk sugar evidently has a different reducing capacity from dextrose and levulose. In the case of the Leach-Babcock test for fat one can readily see that errors can creep in while drawing off the supernatant fluid in three instances.

E. B. Holland, Massachusetts station: Solution: A 20 per cent solution was prepared by diluting 100 grams of the milk to 500 cc. There was some separation of curd or fat, presumably the latter, which must have vitiated the results to some extent.

Moisture: Several aliquots of 5 and 10 cc were evaporated on quartz sand in a flat-bottomed dish at a low temperature until the bulk of the water was expelled, then

dried in an electric oven for two hours at 100° C. or in a vacuum oven below 70° C. (gauge reading, 29 inches).

Evaporation in a flat-bottomed dish without sand yielded low results, probably due to a retention of moisture by the nitrogenous film which formed on the surface. Similar treatment in a vacuum oven gave higher results, but below that obtained on sand. These figures are not reported.

Ash: Twenty-five cc were evaporated in a platinum dish with 5 cc of concentrated nitric acid and burned to a white ash.

Protein: Ten cc were treated by the Kjeldahl-Gunning method.

Fat: Our work of last season indicated that a single extraction gave higher results than the double and saved time and work. The dried solids on the sand were pulverized, washed with water, dried, and extracted with dry ethyl ether in a continuous extractor. Long heating of the residue containing fat (at 100° C.) should be avoided, as it appears to reduce the amount of fat that can be extracted.

C. H. Jones, Vermont station: The result reported on fat by the modified Babcock method is the average of ten determinations on three distinct 40 per cent solutions. The individual readings were 2.60, 2.65, 2.80, and seven readings of 2.70. Lactose determinations were made on distinct portions of the original sample. In the sucrose determination it was necessary to dilute the solution after inversion to 200 cc in order to have an excess of copper in the Fehling solution.

The result reported by the Leach method is the average of two determinations, 70.70 and 70.58 per cent, respectively. The platinum dish used did not have an absolutely flat bottom, but it was the nearest approach to anything of the kind available. I am at present unable to explain the difference obtained by the two methods, unless a too complete drying and consequent breaking down is obtained with Method A.

The result reported on the double-extraction method for fat is the average of two determinations from distinct solutions. They were, respectively, 8.20 and 8.12 per cent. The only awkward feature is the size of the filter paper used. The following modification of the Babcock method described was suggested by the use of the hardened filter on other laboratory determinations:

Method: Place 15 cc of the 40 per cent solution (6 grams) in a small-lipped beaker, diameter 1.5 inches, height 2 inches. Dilute with an equal amount of water; add 4 cc Fehling's copper solution; stir with glass rod. Filter through a 12.5 cm C. S. and S. 575 hardened filter. Wash thoroughly with water; stir on the filter with glass rod (100 cc is usually enough, though 160 cc had no lowering effect on the result). Return precipitate to original beaker, removing any remaining particles by washing with hot water through a fine-jet wash bottle. (The bulk is easily kept below 17 cc.) Stir with a glass rod. Pour into Babcock milk bottle. Add a portion of the acid to the residue in the beaker. Mix thoroughly, using stirring rod. Transfer to Babcock bottle. Repeat with remainder of acid. *Shake milk bottle thoroughly*, and then rinse beaker with a little hot water from the wash bottle and put into test bottle. Run as usual. The individual results obtained by this procedure were 2.70 for five readings and 2.65 on the sixth, three different 40 per cent solutions used.

I find it desirable, both with this method and with Method B, to use rather more sulphuric acid than is specified; often 18.5 cc. While the results by this procedure are not different from those obtained by Method B, yet when a number of samples are run a considerable gain is made in actual working time.

DISCUSSION OF RESULTS.

The results obtained this year are quite satisfactory on all determinations except fat. The lactose results, with three exceptions, are probably as good as one could hope to get from a number of chemists working independently and not making a specialty of sugar determinations. There are some variations in total solids for which it seems difficult to account. One might think it due to variations in the different cans of milk were it not for the fact that in some cases when the solids were as much as 2 per cent low, the other determinations, such as proteids, ash, etc., were as high or higher than the average. The referee can only account for these discrepancies in one of two ways, faulty sampling, or that the sugars were allowed to ferment and cause loss before the determinations were made. It is believed that all determinations should be made as soon as the solutions are made up, and no solution which has stood in a warm laboratory twenty-four hours should be used for the determinations of solids or sugars. Leach's method appears to give high results, probably because of the large amount of sugar present to hold the water.

The ash results are for the most part very good and concordant, with the exception of three, which were probably burned down hard without leaching. It is very evident that leaching with hot water after thorough charring is necessary in the presence of sucrose.

The sucrose results are probably as good as one could expect to obtain, but inasmuch as this is not a normal but an added constituent of the milk it is best determined by difference.

The results on fat, one of the most important constituents of the milk, are far from being satisfactory. The referee believes that these discrepancies are due to three causes. First, lack of experience with this kind of material. Second, a lack of detail in giving the double-extraction method in Circular 32, and, third, faulty instructions in directing a 40 per cent solution to be used for this method. This solution, as shown by the tables below, is too concentrated to get the best results when as large an amount as 40 per cent cane sugar is present. This degree of concentration makes such a thick layer of sugar on the paper coil that quite a large proportion of the fat is left on the paper after the first extraction, and then soaking in water causes a mechanical loss of fat when the sugar is dissolved off. Such loss was proven by examination of the slight scum rising on the water, which, under the microscope, showed the presence of fat globules. When the work is carefully done, however, and dry ether is used, this loss is only small, amounting to one or two tenths of 1 per cent. Our results show that as high results can be obtained with 1 gram in a 10 or 20 per cent solution in a single extraction of fourteen hours as with a more concentrated 40 per cent solution and a double extraction. The highest and most concordant results, however, were obtained with a 10 or 20 per cent solution and double extraction.

Mr. Geisler, the originator of the double-extraction method, stated, under date of January, 1908, that he had no changes to suggest from those given in his original paper published in the *Journal of the American Chemical Society* in 1900 except that the time of each extraction should be extended to seven or eight hours instead of four or five and that strictly dry ether probably is the best solvent on account of its constant boiling point. In Mr. Geisler's paper he emphasizes the fact that the milk should be evenly distributed over the surface of the paper coil; also that the ether should be anhydrous to prevent the paper coil from becoming soggy. The method of drying on asbestos or paper in tubes is not so desirable as on strips of paper on account of the long heating required to dry out completely.

The modified Babcock method, in the hands of men who have had much experience with it, usually gives very good results, but it is not to be considered as accurate as the gravimetric, and as the reading is multiplied by three every error is increased threefold. When the milk was fresh we were able to get very clear separations with the copper sulphate in the centrifuge, and the sugar solution could be easily decanted without loss of fat, the curd and fat remaining in a hard mass at the bottom of the bottle, but in testing some cans later, which had stood in the laboratory for three or four months, the separation was not so complete, and it was necessary to pass the solution through a filter, washing the particles of curd back into the bottle to obtain all the fat, and in several instances the results were low. This operation made the method somewhat longer and more tedious.

The results reported by the Gottlieb method are not very satisfactory and the referee found but little time to test it. Only two tests were made and they gave by direct weight over 9 per cent fat, but it was found that some proteid or foreign matter was clinging to the bottom of the flasks after the fat was dissolved out with hot benzene. After weighing and deducing this weight from the original the results agreed very closely with the gravimetric. The writer believes that by observing proper precautions this method can be made very reliable, particularly for sweetened condensed milk. Small separatory funnels are more desirable than the long tubes recommended in the method.

Some figures obtained in the Maine laboratory are given in the following table, and lead the writer to believe that good results can be obtained when the double-extraction method is carried out as follows:

Prepare strips of soft white filter paper 4 by 24 inches of the quality of the S. & S. No. 597, by soaking two or three hours in alcohol, then after thoroughly drying in the oven extracting several hours with ether until no residue is left from the ether as it comes through. Then take 10 cc of a 20 per cent solution of the condensed milk and distribute it carefully over the whole surface of the thoroughly dried paper. This is best done by attaching one end of the paper to some object and holding the other end out straight so that the pipette can be emptied by passing the point back and forth over the whole surface. To dry the paper, suspend it over a copper wire in the drying oven, where it will thoroughly dry out in two hours or much more rapidly than if coiled up or put in a tube. After drying roll up in a coil, wind with thread or small copper wire, place in the extractor, and extract not less than eight hours. If it is the sweetened product remove the coils from the extractor, loosen the wire or thread, dry and suspend in 500 cc of water for two hours, then return the coils to the oven and dry as before, and extract again for not less than five hours. Five cc of milk and a coil 4 by 12 inches may be used if preferred.

Determination of fat in condensed milk by modifications of the double-extraction method.

Modifications.	Fat.
	<i>Per cent.</i>
5 cc (2 grams) of 40 per cent solution extracted 8 hours with ether:	
Exhausted with water and extracted 5 hours more (average of 4 samples).....	8.28
Exhausted a second time and extracted a third time.....	8.45
5 cc (2 grams) of a 20 per cent solution on a coil 5 by 24 inches:	
10 Extracted 10 hours.....	8.55
Exhausted with water and extracted 5 hours more.....	8.78
5 cc (2 grams) of a 20 per cent solution on a coil 5 by 24 inches:	
Extracted 14 hours.....	8.42
Exhausted with water and extracted again 6 hours.....	8.69
5 cc (1 gram) of a 20 per cent solution on a coil 5 by 12 inches:	
Extracted 5 hours.....	8.45
Exhausted with water and extracted 5 hours more.....	8.78
5 cc (1 gram) of a 20 per cent solution on a coil 5 by 12 inches:	
Extracted 10 hours with ether.....	8.36
Exhausted with water and extracted again.....	8.85
10 cc (1 gram) of a 10 per cent solution on a coil 5 by 24 inches:	
Extracted with ether 14 hours.....	8.42
Exhausted with water and extracted 5 hours more.....	8.95

RECOMMENDATIONS.

It is recommended that—

(1) The following methods be adopted as official methods:

1. PREPARATION OF SAMPLE.

Place the can, if cold, in water at 30° to 35° C. until warm. Open and mix thoroughly by transferring the contents of the can to some dish sufficiently large to thoroughly stir and make the whole mass homogeneous. Care must be taken to scrape out all milk adhering to the interior of the can. Weigh 100 grams into a 500 cc flask and make up to the mark with water. If the milk will not completely dissolve, each portion must be weighed out separately for analysis.

2. TOTAL SOLIDS.

Use 10 cc of the above 20 per cent solution and proceed as in the case of milk analysis according to the method given in Bulletin 107, page 117, Method I, drying either on sand or asbestos fiber.

3. ASH.

Ignite the residue from 10 cc of the 20 per cent solution at low red heat, leach with hot water if sucrose is present, ignite the residue and filter until white, add the leachings, evaporate to dryness again in usual manner and weigh.

4. PROTEIN.

Determine nitrogen by Kjeldahl or Gunning method in 10 cc of the 20 per cent solution and multiply by 6.38.

5. LACTOSE.

Dilute 100 cc of the 20 per cent solution in a 250 cc flask to about 200 cc; add 6 cc of Fehling's copper sulphate solution and make up to the mark; filter through a dry filter and determine lactose by the Walker method, boiling only two minutes with the Fehling solution.

(2) The methods for determining sucrose and fat be given further study.

REPORT ON FOODS AND FEEDING STUFFS.

By FRED W. MORSE, *Associate Referee*.

The request to serve as referee on cattle foods was unexpected and found me unfamiliar with the most recent work of the association on this class of materials. Noting that it was recommended last year to continue the trial of the methyl pentosan determination after the method of Ellett, an attempt was made to simplify the method before asking for cooperation from other members. Wholly satisfactory results have not yet been obtained, but it seems possible, with a little more time, to accomplish such a modification.

It was also planned to compare the effects of the use of Ellett's method on some standard cattle food alongside of a substance known to contain methyl pentosan. For the latter there was accessible plenty of the seaweed, *Fucus vesiculosus* or rockweed, and a quantity was obtained, dried, and pulverized. For the standard cattle food, wheat bran was selected, since its content of pentosan is good, and Widtsoe reported no evidence of methyl furfural in it by the qualitative tests.

The method of procedure was to follow the provisional method for pentosan determination throughout and, after weighing the precipitated phloroglucid, to extract with alcohol by Ellett's method of digesting the crucible and contents in a small quantity of alcohol at 65° C., filtering, and repeating the operation until the filtrate finally becomes colorless. A marked solubility of the precipitate was observed in both cases. This was unexpected in the bran, and considerable time was spent in repeating determinations. Results on bran varied much; but the seaweed gave reasonably concordant figures. By this time it was too late to send out samples to other chemists. Another point was noticed in the prosecution of the work, namely, that the provisional method for pentosans seldom if ever yielded furfural-free distillates when the prescribed limit of volume was reached. The drops would still show traces of furfural.

These points of disagreement from published matter about the different forms of pentosans have convinced the referee that more work is needed on this provisional method in some of the details. There is an important field for research in our common coarse fodders and the concentrated by-products in working out the constituents of the nitrogen-free extract. Most of the methods now in use are difficult of manipulation and more or less approximate in their results. Comparatively little attention is paid to them, since the conventional methods of fodder analysis answer the practical feeder's purpose.

Nevertheless, progress in nutrition studies demands more attention to the less-known carbohydrates, since their digestibility and consequent food value are unknown quantities.

The referee has no recommendations to make; but if no instructions are received from the association it is his intention to continue the study of these newest methods of determining the less-known carbohydrates.

Ellett's method applied to Fucus vesiculosus.

[Two grams of material.]

Total phloro- glucid.	Loss by ex- traction with alcohol.
<i>Gram.</i>	<i>Gram.</i>
0.2403	0.0429
.2467	.0516
.2497	.0341
.2405	.0434
.2664	.0446
Mean .2487	.0433

BEST RESULTS BY WASHING WITH HOT ALCOHOL.

<i>Gram.</i>	<i>Gram.</i>
0.2366	0.0407
.2533	.0366

THE DETERMINATION OF ACIDITY IN CATTLE FEEDS.By JOHN PHILLIPS STREET, *Referee.*

The acidity of a cattle food is due to the presence of hydrogen ions. In a solution containing a mixture of salts of organic and inorganic acids it makes practically no difference whether this acidity was originally produced by the addition of a small amount of an organic or of an inorganic acid, for the final result is essentially the same; that is, the presence of a certain proportion of free hydrogen ions and of the ions of all the various salts which are present in the solution. The question of acidity, therefore, is one of degree rather than of kind and, from a physiological view point, depends on the nature of the salts which are present in the solution under consideration. Let us take an example. We have a solution containing sodium chlorid and sodium acetate. In this we have sodium ions, chlorin ions, acetate ions, undissociated sodium acetate molecules, and undissociated sodium chlorid molecules. If a small quantity of hydrochloric acid is added to this solution it will then contain, in addition to the substances above named, a certain quantity of hydrogen ions and a correspondingly greater quantity of chlorin ions. If a molecularly equivalent quantity of acetic acid is added instead of hydrochloric, the solution will contain the hydrogen ions, as in the first case, and the number of acetate ions will be correspondingly increased.

If we now measure the acidity of each of these two solutions with phenolphthalein as an indicator, the result will be the same, for this indicator gives a pretty accurate measure of free and potentially free hydrogen ions. If we measure the acidity of these solutions by means of delicate litmus paper the degree of acidity will be found to be less than that as determined by phenolphthalein. The reason for this is to be found in the fact that litmus is a relatively stronger acid than phenolphthalein and reacts with the base before all the acid hydrogen of the acetic acid has been acted on. The effect of the presence of organic salts is to reduce the number of free hydrogen ions, in comparison with that which would be present in a solution to which had been added the same quantity of mineral acid in the presence simply of inorganic salts of strong bases with strong acids, such as sodium chlorid or sodium sulphate, and it is also clear that it is not possible to determine in a solution containing a mixture of organic and inorganic salts, which show an acid reaction, whether this reaction was originally caused by the action of a mineral acid or of an organic acid. The indicators that are commonly supposed to distinguish between mineral and organic acids in mixtures

containing salts of weak bases and strong acids, or of strong bases and weak acids, give entirely different results from those obtained in solutions free from such salts, for by hydrolytic dissociation these salts contribute to the solution a certain quantity of hydrogen or hydroxyl ions, according to the nature of the salts present and the concentration of the solution, which ions exert an effect on the indicator in one direction or the other.

In a mixture of weak and strong acids and their salts, phenolphthalein, which is the weakest indicator commonly employed, gives the total amount of acids present stronger than phenolphthalein, itself an exceedingly weak acid. If a stronger acid indicator, e. g., litmus, is in the above mixture, it will appear to have less total acid, because the litmus itself reacts with the base before the weaker acids are acted on.

It is thus clear that, for such a mixture, it is impossible to determine the acidity of its solution, and, furthermore, it is not even possible by titration to determine the actual concentration in free hydrogen ions, which, from a physiological standpoint, is the true question under consideration.

It has been the practice of physical chemists to determine the concentration in hydrogen ions by inverting cane sugar, a process which closely corresponds with the enzym reactions of physiological processes.

The acidity of a cattle feed may come from a mineral acid used in its preparation, from organic acids natural to the product itself, or developed by fermentation during its preparation, and possibly, in some cases, from phosphates having an acid reaction and normally present in the feed.

Jordan's studies (Bulletin 238, Geneva station), however, indicate that "our commercial feeds of vegetable origin do not contain appreciable quantities of phosphorus in inorganic combination."

Ensilage is an example of a feed containing considerable amounts of organic acids developed in the silo by fermentation. A number of other feeds which are by-products of manufacturing processes contain organic acids resulting, likewise, from fermentations taking place during manufacture.

In view of the fact that the presence of free mineral acids in certain feeds has been suspected or affirmed, I wish to raise the question of the methods involved and ask the association to make it the subject of inquiry, in order that an accurate method of testing for acidity may be found and adopted. As matters now stand, we are depending wholly on volumetric methods and the use of indicators, and the question to be settled first of all is, Just what do indicators indicate?

Too little attention has been given to the acidity due to the proteins themselves and their varying action with different indicators. Osborne ^a has pointed out that the proteins are not neutral bodies, like the carbohydrates, and that the general assumption that a solution containing protein matter, and showing neither acid nor alkaline with litmus, is chemically neutral, is erroneous. Many experiments have shown that certain protein solutions, when neutral to litmus, are acid to phenolphthalein and alkaline to lacmoid. It has also been established that a notable amount of acid can be added to a protein solution before an acid reaction with tropaeolin, alizarin, or phloroglucin and vanillin is given.

As Osborne says, ^b it is of importance "to know whether litmus can be used to determine the point when all combined acid has been converted into neutral salts of potassium or sodium and all the protein substance has been set free, or whether, as we know is the case, when tropaeolin or lacmoid is used as an indicator, more acid still remains combined."

Aqueous solutions of crystallized ovalbumin, solutions in sodium chlorid brine of excelsin, amandin, vignin, conglutin, glycinin, corylin, phaseolin, and legumin, and solutions of zein, gliadin, and hordein in 75 to 90 per cent alcohol, which were either

^a J. Amer. Chem. Soc., 1902, 24: 39.

^b Loc. cit.

neutral or acid to sensitive neutral litmus paper, when made neutral to litmus were, in every case, still acid toward phenolphthalein.

Osborne further says:

To render gram portions of these several protein preparations neutral to litmus required in a few cases not any, in most cases from 0.1 to 1.5 cc of decinormal alkali; while to make the same gram portions neutral to phenolphthalein required the further addition of from 0.7 to 1 cc of decinormal alkali, except for legumin, which required 2 cc. Edestin made neutral to phenolphthalein and dissolved in sodium chlorid solution reacts distinctly alkaline toward litmus. This alkaline reaction is caused by the edestin itself and not by organic salts of the alkali, since such preparations yield a very small amount of ash, less than 0.05 per cent, which is neutral to both litmus and phenolphthalein. * * * Solutions of all the other protein bodies I have examined, when similarly made neutral to phenolphthalein, react decidedly alkaline with litmus.

In the investigation which the referee reports at this time no attempt was made to determine total acidity, only those acids being taken into account which were extracted by a rather prolonged treatment with water. Sixteen samples of gluten feed, representing five brands, two of wheat bran and one each of wheat middlings, wheat feed and cottonseed meal, were examined.

Ten grams of the feed were weighed into a beaker and stirred with 50 cc of water for ten minutes, then transferred to a plain wet filter and washed with successive small portions of water, until the washings amounted to 150 cc; the extract was then made up to 200 cc with water and 20 cc portions (=1 gram feed) used in the subsequent titrations. A blank determination was also made with 200 cc of water run through a filter paper as before, and the washings were found to be neutral to methyl orange, phenolphthalein, and litmus.

The following indicators were used:

Phenolphthalein: One gram dissolved in 100 cc of 50 per cent alcohol.

Litmus paper: Very sensitive neutral paper.

Methyl orange: One gram dissolved in 1,000 cc of water.

Congo red: One gram dissolved in 100 cc of 30 per cent alcohol.

Günzburg's reagent: Two grams of phloroglucin and 1 gram of vanillin dissolved in 30 grams of alcohol.

Toeffer's reagent: One per cent solution of phenolphthalein in alcohol and 0.5 per cent solution of dimethylamidoazobenzol.

The alkali used was approximately decinormal sodium hydroxid, 1 cc being equal to 0.003996 gram sodium hydroxid.

Twenty cubic centimeter portions of the watery extract, equal to 1 gram of feed, were taken for each test. Owing to the usually highly colored solutions, the aliquot was diluted with 500 cc of water for the test with methyl orange, and with 50 cc for the other indicators, except in the Günzburg test, where 0.5 cc of the extract and the same quantity of the reagent were used. Three hydrochloric acid solutions were also prepared, N/14, N/28, and N/56, respectively.

The Günzburg and Toeffer tests and Congo Red are recommended as reliable in determining whether or not free mineral acid is present. These tests were applied first to the three hydrochloric acid solutions and unmistakable positive results were secured with all, the most dilute acid used, N/56, equivalent to about 0.065 per cent of hydrochloric acid, responding perfectly to the reactions indicated. A mixture of one of the aqueous feed extracts and dilute hydrochloric acid, the total mixture containing 0.065 per cent of free hydrochloric acid, was likewise subjected to these tests and positive proof was secured that nothing present in the feed extract in any way interfered with the delicacy of the reactions. However, following the suggestions of Osborne's work, when the salt of a weak acid, for instance, sodium acetate, was added to these same test solutions, none of the above prescribed tests for free mineral acidity responded, although hydrochloric acid had been added in every case. This experiment shows quite conclusively the danger and inaccuracy of asserting either the presence or absence of mineral acids from data obtained by these tests.

None of the feed extracts showed any acidity to methyl orange, a result quite to be expected. Referring to methyl orange, Ostwald says ^a "should the acid be weak, i. e., only slightly ionizable (the ionization being still further reduced by the presence of the neutral salt formed in the liquid), the quantity of hydrogen ions on passing the point of neutralization is too small to allow of the formation of a visible amount of the nonionized molecules of methyl orange, and the red color only appears after a considerable excess has been added, and then only by degrees."

The conditions thus described by Ostwald seem to be identical with what we have in these feed extracts. Five samples of Buffalo gluten feed required from 1.10 to 1.80 cc of decinormal alkali to neutralize the acidity of 1 gram of feed, using litmus; from 2.55 to 3.40 cc, using phenolphthalein, and from 2.70 to 3.65 cc, using Toepfer's reagent.

Five samples of Globe gluten feed, with the same amount of feed, required from 1.35 to 1.90 cc with litmus; 2.95 to 3.80 cc with phenolphthalein and from 2.90 to 3.80 cc with Toepfer's reagent.

Cream of corn gluten feed and Pekin gluten feed gave corresponding results with the three indicators; while Douglas gluten feed showed the same relation, but a much lower acidity, 0.25 cc with litmus, 0.40 cc with phenolphthalein and 0.35 with Toepfer's reagent.

The samples of wheat bran, middlings, and feed required 0.50 to 1 cc with litmus, 0.90 to 1.70 with phenolphthalein and 1 to 1.80 with Toepfer's reagent.

Cottonseed meal required 0.65 cc, 1 cc and 1 cc, respectively, with the three indicators.

The average acidity of all the feeds was 1.18 cc with litmus, 2.37 cc with phenolphthalein and 2.50 cc with Toepfer's reagent.

These results correspond perfectly with what we should expect if the acidity came from dissociation of protein salts alone, or from salts of weak organic acids. I therefore urge the association to take up the matter of acidity of cattle feeds and consider how the results obtained by current methods can be applied to agricultural problems.

The appended table presents the determinations in detail.

Acidity of gluten feeds.

[In terms of 1 gram of feed.]

Num- ber.	Brand.	Protein.	Phenolphthalein.		Litmus.		Toepfer test (dimethylamidoazobenzol and phenolphthalein).	
			Tenth-normal sodium hydroxid.	Equal to grams of sodium hydroxid.	Tenth-normal sodium hydroxid.	Equal to grams of sodium hydroxid.	Tenth-normal sodium hydroxid.	Equal to grams of sodium hydroxid.
		<i>Per cent.</i>	<i>cc.</i>		<i>cc.</i>		<i>cc.</i>	
21435	Buffalo.....	23.37	3.40	0.0136	1.80	0.0072	3.50	0.0140
21448	do.....	26.62	2.55	.0102	1.20	.0048	3.10	.0124
21454	do.....	26.06	3.10	.0124	1.60	.0064	3.65	.0146
21470	do.....	26.31	3.35	.0134	1.65	.0066	3.65	.0146
21548	do.....	25.00	2.70	.0108	1.10	.0044	2.70	.0108
21456	Cream of corn.....	24.75	2.60	.0104	1.20	.0048	3.00	.0120
21518	Douglas.....	20.12	.40	.0016	.25	.0010	.35	.0014
21416	Globe.....	25.87	3.55	.0142	1.85	.0074	3.80	.0152
21439	do.....	26.00	3.40	.0136	1.85	.0074	3.50	.0140
21490	do.....	26.94	3.80	.0152	1.90	.0076	3.70	.0148
21502	do.....	26.37	2.95	.0118	1.45	.0058	2.90	.0116
21549	do.....	25.06	3.40	.0136	1.35	.0054	3.45	.0138
21469	Pekin.....	26.75	2.85	.0114	1.70	.0068	3.20	.0128
21487	Brand unknown.....	28.19	3.65	.0146	1.60	.0064	3.80	.0152
21516	do.....	22.75	.50	.0020	.40	.0016	.70	.0028
21553	do.....	21.62	1.05	.0042	.60	.0024	1.30	.0052
21415	Wheat bran.....	15.31	1.00	.0040	.50	.0020	1.30	.0052
21458	do.....	14.81	1.00	.0040	.50	.0020	1.10	.0044
21423	Wheat middlings.....	18.06	1.70	.0068	1.00	.0040	1.80	.0072
21437	Wheat feed.....	15.94	.90	.0036	.60	.0024	1.00	.0040
21428	Cottonseed meal.....	45.06	1.00	.0040	.65	.0026	1.00	.0040

THE MANUFACTURE OF GLUTEN FEED.

By T. B. WAGNER.

Among the concentrated feeding stuffs found on the American market we may concede to gluten feed the first place, not only because of the high percentage of nutritive materials in gluten feed, but because of its palatability and its remarkable degree of digestibility. Within the last few months various statements have appeared in chemical journals, as well as in bulletins issued by agricultural experiment stations, with reference to the chemical analysis of this product. Considering the importance of gluten feed as an animal food, anything published on the subject will be read with interest, not only by officials connected with agricultural experiment stations, but by dealers and buyers, and, last but not least, by the manufacturers themselves. In view of this general interest, it may not be amiss to state the details of its manufacture. Broadly speaking, gluten feed is the ground kernel of Indian corn, from which the germ and most of the starch have been removed. The following steps lead up to its production:

The corn bought by us is of the No. 2 and No. 3 grades. To remove impurities, stones, dirt, dust, etc., the grain is passed through cleaning and separating machinery and the purified corn is then delivered to the steeping tanks, wherein it is soaked in warm water, slightly acidulated with sulphur dioxide. This treatment brings about a softening of the grain and facilitates the subsequent separation of the germ, which is effected after it has passed through a preliminary grinding whereby the corn is broken up and the germ set free. The balance of the material is now ground fine in Buhr mills, the coarser part, namely, the bran, being separated by running the mass over silk sieves, while the starch liquor is concentrated and sent over slightly inclined planes, the "starch tables," upon which, by a process of settlement and washing, the starch fills up in a solid layer. The lighter ingredients, gluten, fiber, etc., are carried off in the current of water over the end of the starch tables. We have thus obtained, first, the germ from which the well-known corn oil and corn-oil cake are obtained; second, the starch which furnishes the raw material for the corn starch of commerce and the manufacture of corn sirup and corn sugar; third, the bran, being the hulls of the kernel; and, fourth, the gluten. The third and fourth, after repeated washings, are united, when still in a wet state, deprived of the largest part of the water by filter pressing, and delivered to the driers, where the water is reduced to approximately 10 per cent. The feed is now passed through grinding mills and reduced to a considerable degree of fineness.

The gluten feed thus obtained varies in composition in proportion to the efficiency factor prevailing in the individual works. For instance, in a well equipped and well regulated factory the amount of protein usually runs at 26 per cent (on a commercial basis), whereas in factories conducted less efficiently the amount of protein may not exceed 18 per cent. The amount of starch in the feed will vary correspondingly. I have made the statement before that gluten feed represents the corn minus germ and starch. You will ask, and very properly so, What becomes of the mineral constituents of the corn and the soluble organic matter, which are extremely valuable—as, for instance, the organic phosphorus compounds? By far the largest amount of these constituents is leached out in the steeping of the corn. Were it desired only to recover the phosphorus salts, there would not be much difficulty involved in isolating them, but the steep water contains a large amount of other ingredients which greatly add to the food value of the gluten feed, such as albuminoids, sugar and other carbohydrates, potassium salts, etc., which, however, are hygroscopic and frustrate all efforts to recover them in dry form. Dr. Arno Behr devised ways and means of recovering these substances, which are fully described in United States patent No. 491,234, issued February 7. 1893. Briefly explained, Behr recovers these constituents of the

corn by evaporating the steep water after careful treatment to a thick sirup, which contains these substances partly in solution and partly in suspension. This sirup is added to the feed, which latter forms an ideal absorbent.

An analysis of gluten feed thus prepared has the following average composition:

	Per cent.
Water.....	10.36
Protein.....	25.95
Fat.....	2.18
Starch.....	18.09
Fiber.....	6.50
Ash.....	3.70
Nitrogen-free substance (by difference) ^a	33.22
Soluble (approximate).....	15.50

From the process outlined above it is obvious that no extraneous matter is introduced into the feed and that the ingredients which go to make up the feed occur there in the same form as in the corn itself, although, of course, in a more concentrated form. It was not without surprise, therefore, that I noticed in a number of analyses published recently a reference to an "acidity" of the feed, which was reported as hydrochloric acid. It is not quite plain why the acidity was expressed in such a manner, as no hydrochloric acid, or for that matter any other mineral acid, is present, none having been introduced at any stage of the process of manufacture. It certainly would not occur to anyone to report the acidity in fruits, vegetables, cider, or wines as hydrochloric acid, no more than in the case of wheat flour—patent flour—in which the acidity runs nearly as high as some of the acidities reported in gluten feeds. I am not prepared to state at this moment with any degree of finality whether this apparent acidity is due to acid salts, such as the organic phosphorus compounds, or to the presence of a slight amount of lactic acid, or to proteid bodies, such as the acid albumins; but, whatever causes it may be due to, if the absence of free mineral acids has been proved, it should be reported as an organic acid, preferably lactic. It might perhaps be still better to state the number of cubic centimeters of normal alkali required to neutralize.

In this connection it is of import to note the varying results obtained in acidity determinations, depending upon the character of the indicator employed. To cite an instance, we have found that phenolphthalein causes the acidity to appear two to three times higher than rosolic acid. Again, when methyl orange is used, an alkalinity is indicated. These discrepancies and variations make it desirable—in fact, necessary—that the official methods governing the analyses of feeding stuffs provide a standard indicator for such acidity; means should also be provided for expressing properly such acidity as may be found in the feed.

If, as a safeguard, it is deemed advisable to test for free mineral acid, Toepfer's test (dimethylamidoazobenzol), or the even more delicate Günzburg test (phloroglucin), are to be recommended. These tests are generally employed in physiological research and reveal the slightest traces of free mineral acids in the presence of organic acids.

A great desideratum in gluten feed is uniformity; that is, the feed made at the different factories located in different sections of the country should be uniform in composition as well as in appearance. So far as the first is concerned, the variation is very slight, the processes employed in our various factories being under such control as to insure practically uniform composition, irrespective of point of manufacture.

The appearance of the feed is of considerable moment. In former years the corn delivered to our factories was mostly of the yellow type, the amount of white corn delivered being rather insignificant. During the past four years, however, the situa-

^a Contains 17.18 per cent pentosans.

tion has been quite the reverse. The supply of corn is not within our control. We have accomplished uniformity in our feeds so far as protein, fat, and the other ingredients are concerned, and so far as the physical condition of the feed is involved, but we can not reach the same degree of uniformity as regards color so long as the selection of the corn is not within our power. Gluten feed obtained exclusively from yellow corn has a beautiful yellow color, whereas feed made from white corn has an uninviting grayish color, so that, depending upon the amount of yellow and white corn going through the process, the color of the resultant feed may vary from a golden yellow through all the hues down to a grayish white. You will recognize the difficulties connected with the marketing of a product which to-day may run yellow and a week from now white. Speaking from my own experience, this point was brought home to me very forcibly in 1904, when the white variety of corn predominated in our corn supply. The feed produced from such corn was uninviting in appearance. In a very short time dealers, particularly in the Eastern States, began to complain, stating that they were not receiving the old standard gluten feed which they had been familiar with for a long period of years. Our assurance that the feed was the same, that the amount of proteid matter was the same, that the feed value was the same, and that the feed was up to standard in every particular, except color, did not avail, and we were not only threatened with, but actually suffered, a considerable loss of business. We advised the trade fully of the existing conditions, emphasis being laid upon the fact that the color should not be the determining factor in fixing the intrinsic or commercial value of the feed. Feeders, however, refused to accept such explanations. It seemed impossible to convince them that a brand of feed, yellow one day and white the next, could have been made by the same methods and be the same feed in fact.

As a solution of this difficulty, it was suggested that wherever the feed ran "short," so far as color was concerned, that the feed be standardized by the addition of the requisite amount of artificial color, preference being given to naphthol yellow-S. The feeder readily accepted this changed condition. Although informed that the feed is artificially colored, he prefers to buy it that way. It is plain from the above that the manufacturer is not acting from choice when adding color to his feed, but he is forced to do so by a popular demand. The practice of standardizing the color of gluten feed is no different than that practiced by the farmer in coloring butter. June butter is his standard, and in adding color to the butter he aims at matching the natural color of June butter, because the consumer likes that particular color best. Thus the feed obtained exclusively from yellow corn is the standard for color, and when a factory receives only two-thirds or less of its supply in the form of yellow corn, sufficient coloring matter is added to match the feed obtained exclusively from yellow corn. It thus happens that at one of our factories, located in southern Illinois, we do not add at this time a grain of color to the feed, whereas in another factory, located in Iowa, color is added in approximately the same proportions as in the case of colored confectionery. In other words, the practice of standardizing the color of the feed is not a regular practice, but depends from day to day entirely upon the character of the corn supply.

As a matter of chemical interest I would like to call attention to the rapidity with which the gluten of the corn combines with azo colors, such as naphthol yellow-S, forming an insoluble lake. This combination is effected without the use of any mordant, acids, or similar agents and tends to prove the acid character of some of the proteid compounds.

REPORT ON THE SEPARATION OF NITROGENOUS BODIES: MILK AND CHEESE PROTEIDS.

By L. L. VAN SLYKE, *Referee*.

The referee selected the following subjects for investigation:

(1) The acetic-acid-precipitation method for determining casein in milk, especially with reference to the following points:

- (a) The use of less acid.
- (b) The influence of acid on the redissolving of casein.
- (c) The effect of temperature on the solution of casein by acetic acid.
- (d) The effect of various preservatives on the accuracy of the acetic-acid method.

(2) The selection of an official method by the association for the determination of milk albumin.

(3) A simple, rapid, and accurate volumetric method for determining milk casein.

For the cooperative work of 1907-8 the referee selected the Matthaiopoulos volumetric method for the determination of casein. Results from only one of the co-operators was received. The method is as follows:

SOLUTIONS REQUIRED.

- (1) An approximately twenty-fifth-normal solution of sulphuric acid.
- (2) A tenth-normal solution of sodium hydroxid.
- (3) A 1 per cent alcoholic solution of phenolphthalein.

METHOD OF PROCEDURE.

Into each of two 200 cc beakers measure 20 cc of milk and 80 cc of water. Call one *A* and the other *B*. Into *A* let the twenty-fifth-normal sulphuric acid run drop by drop with constant stirring of the diluted milk until the casein precipitates in large flakes. After three to five minutes filter through a dry filter (S and S 589, 9 cm, recommended) and collect the filtrate in a graduated, dry, 100 cc flask. If the first portions of the filtrate are turbid, pour back on filter. If the filtrate continues turbid, not enough acid has been used to precipitate the casein completely; in which case take a fresh sample and add 0.2 or 0.3 cc more acid. The amount of acid required varies with different milks, ranging in the sample studied from about 23 to 27.5 cc. Any excess of acid must be avoided. Collect 100 cc of clear filtrate and transfer it to a beaker, rinsing the flask carefully, add 1 cc of the phenolphthalein solution and titrate to a pale pink color with tenth-normal sodium hydroxid. Note the number of cubic centimeters of alkali used.

Treat the contents of beaker *B* with twenty-fifth-normal sulphuric acid, using exactly the same amount as in the case of *A*. Add 1 cc of phenolphthalein solution and titrate to a pale pink with tenth-normal sodium hydroxid. Note the amount of alkali used.

The values obtained in *A* and *B* are then used in making the following calculations:

$$\left[B - \frac{A(100+H)}{100}\right] \times 0.11315,$$

in which *B* is the amount of alkali used in titrating the mixture of water, milk, and twenty-fifth-normal sulphuric acid; *A* is the amount of alkali used in titrating the filtrate; *H* is the amount of twenty-fifth-normal sulphuric acid used in precipitating casein; 0.11315 is a constant factor based on the equivalent weight of casein as shown by its salts with bases. The results are then calculated from 20 to 100 cc.

Each cooperator was requested to apply this method to samples of fresh milk of his own selection and compare the results with those obtained by the official method.

Determination of casein in milk.

Analyst.	Official method.	Volumetric method.
L. W. Fetzer, Maryland station.	2.54	2.73
	2.52	2.79
A. W. Bosworth, New York station.	3.06	3.06
	3.06	3.06
	3.06	3.07
	3.06	3.06
	3.00	2.88
	3.03	2.90
		3.05

From the results thus far obtained, the method appears to be a promising one. It will probably require some modification to give uniform results.

RECOMMENDATION.

It is recommended—

That the referee for 1908–9 be requested to study the following subjects as fully as may be practicable:

- (1) The official method for determining casein as indicated under 1.
- (2) The perfecting of the method for determining milk albumin.

REPORT ON SUGAR.

By A. H. BRYAN, *Referee*, and FRITZ ZERBAN, *Associate Referee*.

The work of the referee and associate referee upon sugar during the past year has been substantially along the lines recommended by the association at its last meeting and has comprised (1) work upon special methods of analysis in their relationship to sugar chemistry; (2) work upon purely chemical methods; and (3) work by a number of collaborators upon methods for the analysis of cane molasses and sugars.

In the investigations of special methods the work has been confined very largely to the study of the application of the refractometer to the estimation of dry substance in the liquid sugar products. This study was published in the *Journal of the American Chemical Society*^a and is not here repeated, but a recommendation based on the work is made.

The associate referee has confined his work mostly to the study of methods of estimating reducing sugars, trying the Monroe-Neubauer crucible (a platinum gooch with a filtering substance of platinum sponge), instead of the ordinary porcelain gooch. The results are given in the *Journal of the American Chemical Society*.^b

The collaborative work consisted of two lines of determinations: (1) Methods of moisture determinations; (2) effect of clarifying agents on the polarization. Two samples were sent out, one of a yellow sugar and the other an open kettle cane molasses. In the circular letter sent out with these samples, the following instructions were given:

INSTRUCTIONS.

- (1) *Moisture on both samples.*
 - (a) Two grams of material on sand to constant weight in vacuum oven at 70° C.
 - (b) Two grams of material without sand to constant weight in vacuum at 70° C.
 - (c) Two grams of sample on sand in water-jacketed oven for ten consecutive hours. Weigh at end of ten hours. Then heat for two-hour intervals until weight is constant.
 - (d) Repeat (c), but do not use sand.

^a 1908, 30: 1443.

^b 1908, 30: 1456.

(e) Two grams of sample on sand in water-jacketed oven for six hours on one day and four hours the following day. Weigh at end of the ten hours. Then heat for two-hour intervals until constant weight is attained.

(f) Same as (e), but do not use sand.

(g) By refractometer. The procedure is the same as for any work with the refractometer. The readings are taken at 28° C. or any other temperature. A few drops of the solution are placed on the prism and the border line adjusted and read as per instructions found in Bulletin 107, page 132. The per cent of water is obtained from table of Geerligs herewith. A table of temperature corrections is also given, so that corrections can be made for any other temperature.

Geerligs's table for dry substance in sugar-house products by the Abbe refractometer, at 28° C.^a

Index.	Per cent dry substance.	Decimals to be added for fractional readings. ^b		Index.	Per cent dry substance.	Decimals to be added for fractional readings. ^b	
1.3335	1	0.0001=0.05	0.0010=0.75	1.4083	45	0.0004=0.2	0.0015=0.75
1.3349	2	0.0002=0.1	0.0011=0.8	1.4104	46	0.0005=0.25	0.0016=0.8
1.3364	3	0.0003=0.2	0.0012=0.8	1.4124	47	0.0006=0.3	0.0017=0.85
1.3379	4	0.0004=0.25	0.0013=0.85	1.4145	48	0.0007=0.35	0.0018=0.9
1.3394	5	0.0005=0.3	0.0014=0.9	1.4166	49	0.0008=0.4	0.0019=0.95
1.3409	6	0.0006=0.4	0.0015=1.0	1.4186	50	0.0009=0.45	0.0020=1.0
1.3424	7	0.0007=0.5		1.4207	51	0.0010=0.5	0.0021=1.0
1.3439	8	0.0008=0.6		1.4228	52	0.0011=0.55	
1.3454	9	0.0009=0.7		1.4249	53		
1.3469	10			1.4270	54		
1.3494	11	0.0001=0.05		1.4292	55	0.0001=0.05	0.0013=0.55
1.3509	12	0.0002=0.1		1.4314	56	0.0002=0.1	0.0014=0.6
1.3516	13	0.0003=0.2		1.4337	57	0.0003=0.1	0.0015=0.65
1.3530	14	0.0004=0.25		1.4359	58	0.0004=0.15	0.0016=0.7
1.3546	15	0.0005=0.3		1.4382	59	0.0005=0.2	0.0017=0.75
1.3562	16	0.0006=0.4		1.4405	60	0.0006=0.25	0.0018=0.8
1.3578	17	0.0007=0.45		1.4428	61	0.0007=0.3	0.0019=0.85
1.3594	18	0.0008=0.5		1.4451	62	0.0008=0.35	0.0020=0.9
1.3611	19	0.0009=0.6		1.4474	63	0.0009=0.4	0.0021=0.9
1.3627	20	0.0010=0.65		1.4497	64	0.0010=0.45	0.0022=0.95
1.3644	21	0.0011=0.7		1.4520	65	0.0011=0.5	0.0023=1.0
1.3661	22	0.0012=0.75		1.4543	66	0.0012=0.5	0.0024=1.0
1.3678	23	0.0013=0.8		1.4567	67		
1.3695	24	0.0014=0.85		1.4591	68		
1.3712	25	0.0015=0.9		1.4615	69		
1.3729	26	0.0016=0.95		1.4639	70		
1.3746	27	0.0001=0.05	0.0012=0.6	1.4663	71		
1.3764	28	0.0002=0.1	0.0013=0.65	1.4687	72		
1.3782	29	0.0003=0.15	0.0014=0.7	1.4711	73	0.0001=0.0	0.0015=0.55
1.3800	30	0.0004=0.2	0.0015=0.75	1.4736	74	0.0002=0.05	0.0016=0.6
1.3818	31	0.0005=0.25	0.0016=0.8	1.4761	75	0.0003=0.1	0.0017=0.65
1.3836	32	0.0006=0.3	0.0017=0.85	1.4786	76	0.0004=0.15	0.0018=0.65
1.3854	33	0.0007=0.35	0.0018=0.9	1.4811	77	0.0005=0.2	0.0019=0.7
1.3872	34	0.0008=0.4	0.0019=0.95	1.4836	78	0.0006=0.2	0.0020=0.75
1.3890	35	0.0009=0.45	0.0020=1.0	1.4862	79	0.0007=0.25	0.0021=0.8
1.3909	36	0.0010=0.5	0.0021=1.0	1.4888	80	0.0008=0.3	0.0022=0.8
1.3928	37	0.0011=0.55		1.4914	81	0.0009=0.35	0.0023=0.85
1.3947	38			1.4940	82	0.0010=0.35	0.0024=0.9
1.3966	39			1.4966	83	0.0011=0.4	0.0025=0.9
1.3984	40			1.4992	84	0.0012=0.45	0.0026=0.95
1.4003	41			1.5019	85	0.0013=0.5	0.0027=1.0
1.4023	42	0.0001=0.05	0.0012=0.6	1.5046	86	0.0014=0.5	0.0028=1.0
1.4043	43	0.0002=0.1	0.0013=0.65	1.5073	87		
1.4063	44	0.0003=0.15	0.0014=0.7	1.5100	88		
				1.5127	89		
				1.5155	90		

^a Intern Sugar J., 10: 69-70.

^b Find in the table the refractive index which is next lower than the reading actually made and note the corresponding whole number for the per cent of dry substance. Subtract the refractive index obtained from the table from the observed reading; the decimal corresponding to this difference, as given in the column so marked, is added to the whole per cent of dry substance as first obtained.

Table of corrections for the temperature.

Dry substance.													
Temperature of the prisms in ° C.	0	5	10	15	20	25	30	40	50	60	70	80	90
Subtract													
20.....	0.53	0.54	0.55	0.56	0.57	0.58	0.60	0.62	0.64	0.62	0.61	0.60	0.58
21.....	.46	.47	.48	.49	.50	.51	.52	.54	.56	.54	.53	.52	.50
22.....	.40	.41	.42	.42	.43	.44	.45	.47	.48	.47	.46	.45	.44
23.....	.33	.33	.34	.35	.36	.37	.38	.39	.40	.39	.38	.38	.38
24.....	.26	.26	.27	.28	.28	.29	.30	.31	.32	.31	.31	.30	.30
25.....	.20	.20	.21	.21	.22	.22	.23	.23	.24	.23	.23	.23	.22
26.....	.12	.12	.13	.14	.14	.15	.15	.16	.16	.16	.15	.15	.14
27.....	.07	.07	.07	.07	.07	.07	.08	.08	.08	.08	.08	.08	.07
Add—													
29.....	0.07	0.07	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.08	0.07
30.....	.12	.12	.13	.14	.14	.14	.15	.15	.16	.16	.16	.15	.14
31.....	.20	.20	.21	.21	.22	.22	.23	.23	.24	.23	.23	.23	.22
32.....	.26	.26	.27	.28	.28	.29	.30	.31	.32	.31	.31	.30	.30
33.....	.33	.33	.34	.35	.36	.37	.38	.39	.40	.39	.38	.38	.38
34.....	.40	.41	.42	.42	.43	.44	.45	.47	.48	.47	.46	.45	.44
35.....	.46	.47	.48	.49	.50	.51	.52	.54	.56	.54	.53	.52	.50

(h) By areometric methods, as found on pages 65-67, Bulletin 107. If time permits it would be well to determine the per cent of water in a number of sugar solutions by methods (g) and also (h) and (b). The comparison being between results of (g) and (b) and (h) and (b). The results could be reported as special samples under (g), giving kind of sirup, also figures obtained by (g), (b), and (h).

(2) *Polarimetric determinations. Effect of various clarifying agents on both samples.*

Weigh out a normal weight and make up to 100 cc, or to such a multiple thereof as may be necessary to secure an accurate polarization, after clarifying as follows:

(a) With lead subacetate solution. (Bull. 46, pp. 38-39; also Bull. 107, p. 40.) Try at least two different quantities of the clarifying agents, reporting the separate polarization.

(b) With normal lead acetate solution. (Saturated solution of lead acetate in water.)

(c) With Horne's dry lead subacetate. (J. Amer. Chem. Soc., 1904, 26 : 186.)

(d) With Herles' solution. No. 1, 250 grams lead nitrate to 500 cc water; No. 2, 25 grams sodium hydroxid to 500 cc water. Use equal parts of each solution, either 5 cc each or up to 10 cc of each. Note whether increased amount changes the polarization.

(e) With alumina cream and hydrosulphite (sodium hydrosulphite, B. A. S. F. or "Blankit"). This with the dry subacetate can be obtained from any of the large dealers in chemical supplies. In this clarification make solution up to required volume, then add a few crystals at a time until decolorization is effected. Polarize at once after filtering and again after standing for some time. Should the solution become cloudy on standing add some kaolin and shake, filter. Also try the following method of procedure: In a solution after clarifying with alumina cream and filtering, just before screwing on cap of polarization tube, add a few crystals of the hydrosulphite and shake. Polarize immediately; note whether on standing there is a change in the polarization.

(f) Invert portions of a, b, c, d, and e and determine the invert reading. Where lead has been used take out the excess with some crystals of potassium oxalate or dry sodium carbonate. Inversion can be accomplished by following (c), page 40, Bulletin 107, or (1), page 39, Bulletin 46, Revised. If the latter reference is used, the equation should read:

$$S = \frac{a-b}{142.66-2} t$$

In this polarization take care to record all temperatures of polarization, dilutions, etc., that results may be compared upon as uniform a basis as possible.

It is also urged that the work on the samples be begun immediately upon their arrival, to avoid changes in composition which might result from fermentation.

A. HUGH BRYAN,
Referee on Sugar.
FRITZ ZERBAN,
Associate Referee.

A number of chemists signified their willingness to cooperate, and reports, in whole or in part, were received from them.

DETERMINATION OF TOTAL SOLIDS.

The work outlined was for the comparison of the vacuum method with the regular method of drying for ten hours. But as ten hours is generally longer than the ordinary laboratory day, a comparison was made of this determination conducted for ten consecutive hours, and also for six hours on one day and four the next. Together with the comparison of methods of determining moisture, the effect of mixing sand with the material to be dried was studied in each case. The refractometer was tried, and the specific gravity was also determined and the moisture calculated.

Determinations of moisture in sugar and molasses.

SUGAR.

Analyst.	Vacuum.		At boiling water temperature.						Refractometer.	Specific gravity.
			10 consecutive hours.		6 and 4 hours.					
	Sand.	No sand.	Sand.	No sand.	Sand.	No sand.				
P. H. Doherty, New Orleans, La.....	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>		
F. W. Liepsner, Washington, D. C.....	a 2.84	a 2.57	Not dry.	2.68 2.90	2.87 Not dry.	2.76 2.70	b 2.60	2.22		
W. P. Naquin, New Orleans, La.....			b 2.10	2.89	2.95	2.97	2.60			
John H. Parkins, Richmond, Va.....			2.68	2.79	2.41	2.37				
F. G. Smith, New Orleans, La.....			2.69	2.74	2.90	2.75	2.60			
R. N. Wilson, Florida.....	c 2.52	c 2.54	2.87	2.48	2.50	2.49				
Average.....	2.68	2.56	2.74	2.75	2.72	2.67	2.60	2.22		

MOLASSES.

P. H. Doherty, New Orleans, La.			^b 28.06	27.67	27.99	27.37	27.04	26.36
F. W. Liepsner, Washington, D. C.	^a 27.04	^a 27.17	27.35	27.48	27.84	27.15	26.27	24.30
W. P. Naquin, New Orleans, La.			27.38	^b 25.55	27.94	27.86	27.04	26.36
J. H. Parkins, Richmond, Va.			27.55	27.26	27.77	27.24	28.50	^d 28.50
F. G. Smith, New Orleans, La.			27.51	27.20	28.19	27.45	27.03	
Average	27.04	27.17	27.45	27.40	27.94	27.41	27.17	25.78

^a Constant at end of 31 hours.

^b Not included in average.

^c Constant at end of 10 hours.

^d By Westphal balance, 23.40.

It is noted here that the ten-hour drying gives higher results than the vacuum method. This is due, no doubt, to a decomposition of the material at the temperature of boiling water. The ten consecutive hour results are lower than when the time is divided. The use of sand plays an important part in the drying, the determinations being higher with sand present. The reason for this is self-evident. The material forms a coating on the sand and between the particles and so presents a larger surface to be affected by the heat. When not used, a hard, dry film forms on

the material and the under layer does not dry. When a small area of liquid is exposed for drying, the amount of moisture going off will be smaller than when a larger surface is exposed. Many chemists prefer and recommend the use of powdered pumice instead of sand. This allows the material to be absorbed. In the referee's opinion, the results so obtained are of no more value than those with the use of sand. Where numerous determinations are to be made, it is an easy matter to wash and clean the sand, while to clean the pumice stone and remove all traces of the sirup is not so easy. Lately the use of a roll of filter paper has been recommended ^a as the absorbent. Wiley (Bureau of Chemistry, Bul. 19, p. 49) recommended that in 1888, but it was thought then to give low results. Mintz by this method reduced the time of drying from seventeen hours to three. This method is practically the method of Adams for milk, and should be given some consideration for next year. Finely flaked asbestos as an absorbent material has been spoken of for drying milk. Browne ^b used it with success in determinations of moisture in apple juices. It is further worthy of trial, since the claim is made that it requires less time for drying than when sand or pumice stone is used. The referee has made a few experiments with the Soxhlet oven, where a current of dry air passes over the material, but the work has not progressed far enough to make a report. A method that bids fair to supersede all others for pure sugar solutions is the use of the refractometer. The comparison of this method made by the collaborators shows its results to be nearer the vacuum results than those of other methods. A second feature of the moisture work was a study of the effect of increasing the time of heating or dryness on the determination. The following table gives the average results obtained by the collaborators:

Determinations of moisture increasing the time of drying.

[Averages based on reports of five collaborators.]

SUGAR.

Modifications of method.	Pre-scribed time of heating.	By heating.						
		12 hours.	14 hours.	16 hours.	18 hours.	20 hours.	22 hours.	24 hours.
On sand:	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
Vacuum.....	2.68							
10 consecutive hours.....	2.58	2.78	2.91	2.89	3.04	3.06		
6 and 4 hours.....	2.72	2.79	2.86	2.92	3.01	3.06		
No sand:								
Vacuum.....	2.56							
10 consecutive hours.....	2.75	2.85	2.87	2.97	3.05	3.00		
6 and 4 hours.....	2.67	2.70	2.81	2.84	2.97	2.97		
Refractometer.....	2.45							

MOLASSES.

On sand:								
Vacuum.....	27.04							
10 consecutive hours.....	27.45	27.86	28.24	28.42	28.50	28.50	28.85	28.88
6 and 4 hours.....	27.94	28.19	28.53	28.67	28.82	28.78	29.02	
No sand:								
Vacuum.....	27.17							
10 consecutive hours.....	27.40	27.43	27.64	27.98	28.20	28.28	28.44	28.58
6 and 4 hours.....	27.41	27.68	27.85	28.15	28.31	28.47	28.58	
Refractometer.....	27.17							

From these figures the importance of not allowing the length of time to exceed ten hours is noted, as active decomposition sets in. This decomposition was greater when sand was used than when it was not, a result one would naturally expect.

^a Centrbl. Zuckerind., 1908, 16 : 1102; Chemical Abstracts, 1908, 2 : 2632.

^b J. Amer. Chem. Soc., 1901, 23 : 873.

EFFECT OF CLARIFICATION AGENTS ON POLARIZATION.

The work carried on was a continuation of that taken up a number of years ago. The clarifying agents studied were neutral lead acetate, subacetate of lead, both wet and dry, Herles' solution or basic lead nitrate, and hydrosulphites. An additional feature was the comparison of the results obtained when using the necessary amount of the precipitant and when using an excess.

The results on the sugar and molasses samples will be considered separately and for a better comparison the results obtained by using the necessary amount of clarifying agent will be discussed. Following this the results of using an excess of clarifying agent will be considered.

Polarizations of sugar with different clarifying agents, using only amount necessary for clarification.

[Normal weight to 100 cc; polarized in 200 mm tube; sucrose factor 142.66.]

SUBACETATE OF LEAD.

Analyst.	Amount of clarifying agent.	Direct polarization.	Corrected invert polarization.	Temperature of polarization.	Sucrose by Clerget method.
	cc.	°V.	°V.	°C.	Per cent.
A. W. Blair, Florida.....	5	93.00	-27.60	28	93.73
P. H. Doherty, New Orleans, La.....	2	93.00	-28.60	27	94.14
J. A. Hall, New York City.....	a 1	92.80	-29.86	25.5	94.43
G. H. Hardin, New York City.....	a 1	92.85	-29.20	27	94.50
W. D. Horne, Yonkers, N. Y.....	b 1	c 92.85		22	c 92.85
W. L. Howells, New Orleans, La.....	d 1	93.40	-26.20	30	93.69
W. P. Naquin, New Orleans, La.....	2	93.22	-27.52	30	94.66
J. H. Parkins, Richmond, Va.....	d 5	c 90.00	-29.04	28	c 92.52
F. G. Smith, New Orleans, La.....	1	93.05	-27.02	27.5	93.14
M. H. Wiley, New York City.....	a 0.5	92.55	-30.03	25.5	94.38
R. N. Wilson, Florida.....	4	93.80	-27.60	27.4	94.14
F. Zerban, New Orleans, La.....	1	92.80	-30.36	26.0	94.99
Average.....		93.05			94.18

DRY SUBACETATE OF LEAD.

	Grams.				
A. W. Blair, Florida.....	0.5	92.85	-27.40	28.0	93.46
P. H. Doherty, New Orleans, La.....	.5	92.60	-29.92	24.5	93.94
J. A. Hall, New York City.....	.5	92.70	-29.86	25.5	94.35
G. H. Hardin, New York City.....	.5	92.70	-29.15	27	94.34
W. D. Horne, Yonkers, N. Y.....	.27	c 92.70	-29.60	22	c 92.44
W. L. Howells, New Orleans, La.....		93.20	-26.10	30	93.45
W. P. Naquin, New Orleans, La.....	.1	92.96	-28.40	29	94.67
J. H. Parkins, Richmond, Va.....		93.40	-28.60	28	94.82
F. G. Smith, New Orleans, La.....		92.89	-27.07	27.5	93.09
M. H. Wiley, New York City.....	.5	92.50	-29.92	25.5	94.23
R. N. Wilson, Florida.....	.5	93.41	-26.49	28.8	93.48
F. Zerban, New Orleans, La.....	1.0	92.80	-29.15	26.5	94.24
Average.....		92.90			94.00

NEUTRAL LEAD ACETATE.

	cc.				
A. W. Blair, Florida.....	2	92.90	-27.40	28	93.58
P. H. Doherty, New Orleans, La.....	3	92.80	-28.60	27	93.98
J. A. Hall, New York City.....	2	92.60	-29.97	25.5	94.28
G. H. Hardin, New York City.....	5	92.80	-29.75	26.0	94.52
W. D. Horne, Yonkers, N. Y.....	c 3.6	92.40		22.0	
W. L. Howells, New Orleans, La.....	1	93.20	-26.50	30	93.77
W. P. Naquin, New Orleans, La.....	3	93.10	-27.85	29.3	94.48
J. H. Parkins, Richmond, Va.....	5	c 92.00	-29.04	28.0	c 94.07
F. G. Smith, New Orleans, La.....	f 1	93.14	-27.30	27.5	93.43
M. H. Wiley, New York City.....	2	92.75	-30.03	25.5	94.38
R. N. Wilson, Florida.....	3	93.58	-27.03	28.8	94.04
F. Zerban, New Orleans, La.....	1	92.90	-28.82	26.5	93.96
Average.....		92.92			94.04

a 54.3 brix.
b 56.0 brix.

c Not included in average.
d 1.25 sp. gr. ,

e 10 per cent solution.
f 20 per cent solution.

Polarizations of sugar with different clarifying agents, using only amount necessary for clarification—Continued.

BASIC LEAD NITRATE (HERLES' SOLUTION).

Analyst.	Amount of clarifying agent.	Direct polarization.	Corrected invert polarization.	Temperature of polarization.	Sucrose by Clerget method.
	cc each.	°V.	°V.	°C.	Per cent.
P. H. Doherty, New Orleans, La.....	2	92.60	-29.70	24.5	93.78
J. A. Hall, New York City.....	2	92.75	-29.86	25.5	94.40
G. H. Hardin, New York City.....	5	92.80	-29.86	26.0	94.60
W. D. Horne, Yonkers, N. Y.....	0.72	92.80			
W. L. Howells, New Orleans, La.....	5	93.50	-25.50	30.0	93.21
W. P. Naquin, New Orleans, La.....	5	93.32	-28.82	28.6	95.16
J. H. Parkins, Richmond, Va.....	5	92.22	-29.70	28.0	94.76
F. G. Smith, New Orleans, La.....	5	93.44		28.5	
M. H. Wiley, New York City.....	5	92.80	-30.14	25.5	94.57
F. Zerban, New Orleans, La.....	1	92.80	-30.47	26.0	95.07
Average.....		92.98			94.39

ALUMINA CREAM AND SODIUM HYDROSULPHITE.

P. H. Doherty, New Orleans, La.....		92.20			
W. D. Horne, Yonkers, N. Y.....	0.7	92.60			
W. L. Howells, New Orleans, La.....		93.10	-25.80	30.0	93.14
J. H. Parkins, Richmond, Va.....		92.00	-30.80	28.0	95.44
F. G. Smith, New Orleans, La.....		93.25	-26.90	29.0	93.75
R. N. Wilson, Florida.....		94.20	-26.83	28.8	94.37
F. Zerban, New Orleans, La.....		92.50	-31.24	26.0	95.43
Average.....		92.75			94.11

* Not included in average.

The results of individual analysts on direct polarization compare very favorably in each method of clarification. There are, however, some higher figures than the average, but in every case the polarization was carried on at a lower temperature or an excess of the precipitant was used. With a few exceptions the results on sucrose by the Clerget method do not differ so widely as the direct polarizations. This difference with the Clerget method is most likely due to the different methods of inverting or to an error in calculation. As a check for the first error it is well to run a test on pure sucrose with each set of determinations. This is especially wise in case of inverting by heat, as the temperature may not be right or the time either too short or too long, and, as a result, either all the sucrose has not been inverted, or the inversion has been carried on so far that reversion products have been formed. Even in the standing method for inversion this blank is valuable in determining the completeness of the inversion. There is a point in the cold inversion that should receive some attention. This is the question of the relation of time and temperature. In a few experiments on the same sugar solution, one inverted by standing at 20° C. for twenty hours showed -12.3, while the other portion by standing at 32° for twenty hours showed -12.08. These figures would make a difference in the Clerget sucrose.

The other point, error in calculation, is one that for some reason or other is rather common. The inversion is carried on by taking 50 cc of the solution and adding 5 cc of acid and not correcting the reading for the increase of 10 per cent in volume. Chemists not using the formula often should guard against this error, as the difference amounts to nearly 3 per cent in high polarizations.

In comparing the average results of direct polarization it is noted that hydrosulphite gives the lowest reading, while wet subacetate gives the highest. The normal acetate and Herles' solution give nearly the same results. Dry subacetate gives readings that are lower than the two above mentioned and stands next to hydrosulphite.

As regards the decolorization effect, Herles' solution equals wet subacetate. The dry subacetate gives solutions a little darker than the above, and next in order is neutral acetate. Hydrosulphite gives a good decolorization, but under certain conditions the solutions become murky from the precipitation of sulphur and also, on standing, the color returns again.

When the precipitation agents are used in excess, the readings are all higher, as shown by the following table. This increase is no doubt due largely to the solution becoming more concentrated from the increased amount of precipitation, and partly also from a change in rotation due to the salts.

Polarizations of sugar with different clarifying agents, using an excess of clarifier.

[Normal weight to 100 cc; polarized in 200 mm tube; sucrose factor 142.66.]

SUBACETATE OF LEAD.

Analyst.	Amount of clarifying agent.	Direct polarization.	Corrected invert polarization.	Temperature of polarization.	Sucrose by Clerget method.
	cc.	* V.	* V.	* C.	Per cent.
P. H. Doherty, New Orleans, La.	3	93.00	-28.60	27	94.14
J. A. Hall, New York City	a 4	92.95	-29.70	25.5	94.42
G. H. Hardin, New York City	a 3	92.95	-29.20	27	94.58
W. D. Horne, Yonkers, N. Y.	b 2	92.90		22	
W. L. Howells, New Orleans, La.	c 2	93.50	-26.00	30	93.60
W. P. Naquin, New Orleans, La.	3	93.24	-27.56	30.3	94.74
J. H. Parkins, Richmond, Va.	c 10	d 91.60	-29.04	28.0	d 93.77
F. G. Smith, New Orleans, La.	2	93.15	-27.30	27.5	93.44
M. H. Wiley, New York City	a 3	92.80	-29.70	25.5	94.30
R. N. Wilson, Florida	6	94.00	-27.00	28.0	94.05
F. Zerban, New Orleans, La.	2	92.80	-30.25	25.5	94.72
	3	92.90	30.14	25.5	94.71
Average		93.11			94.26

DRY LEAD SUBACETATE.

	Grams			
P. H. Doherty, New Orleans, La.	1.00	92.60	-29.70	24.5
W. D. Horne, Yonkers, N. Y.	.75	92.75		
W. P. Naquin, New Orleans, La.	.50	93.26	-28.53	28.8
R. N. Wilson, Florida	1.00	93.61	-26.63	29.0
F. Zerban, New Orleans, La.	2.00	92.50	-29.15	26.5
Average		92.94		

NEUTRAL LEAD ACETATE.

	cc.			
P. H. Doherty, New Orleans, La.	4	92.80	-28.60	27.5
W. P. Naquin, New Orleans, La.	5	93.28	-27.98	29.2
J. H. Parkins, Richmond, Va.	10	d 91.00	-29.04	28.0
F. Zerban, New Orleans, La.	2	92.80	-29.26	26.5
Average		92.96		

BASIC LEAD NITRATE.

	cc each.			
P. H. Doherty, New Orleans, La.	4	92.80	-29.70	25.0
W. L. Howells, New Orleans, La.	10	93.60	-25.50	30.0
W. P. Naquin, New Orleans, La.	10	93.20	-33.22	18.2
F. G. Smith, New Orleans, La.	10	93.55		28.5
F. Zerban, New Orleans, La.	2	93.00	-30.47	26.0
Average		93.23		

a 54.3 brx.

b 56 brx.

c 1.25 sp. gr.

d Not included in average.

The greatest difference is in the Herles' solution, then comes the wet and dry subacetate, which show about the same increase, and the least increase is with normal

acetate. This would naturally be expected, as the Herles' solution forms a precipitate in itself, hence causing concentration, and the excess of wet lead subacetate causes an increase and also a change of precipitate, thereby changing the concentration, while the normal acetate produces no more precipitate with an excess, and hence no change of concentration.

As regards the danger of adding an excess, this is the least in case of the neutral acetate, as an excess is indicated when no more precipitate continues to form. When using wet subacetate a better clarification is reached before the point where more acetate will produce a further precipitation. With dry lead it is difficult to determine when enough has been added. To add by weight takes much time, but where many determinations are to be made varying measures or cups could be used, the weight of the contents having been previously determined. It has the fault of precipitating reducing sugars in as large quantities as the wet subacetate, as noted in last year's report; besides, an excess of this reagent increases the volume of the solution, thereby lowering polarization. This effect is shown in the following experiment: Six hundred cubic centimeters of a solution of pure sucrose were made up and five 100 cc flasks were filled and the following quantities of the dry subacetate were added, shaken, and then polarized, care being taken that the polarization was made at 20° C.

Polarization of pure sucrose solution with varying amounts of dry lead.

Number.	Dry sub- acetate.	Polariza- tion.
	<i>Grams.</i>	<i>° V.</i>
1.....	0.0	81.25
2.....	.5	81.1
3.....	1.0	81.05
4.....	1.5	81.0
5.....	2.0	80.9

It is noted from the table that the polarization has been lowered 0.35° by the addition of 2 grams of the dry lead.

The greater part of the lead subacetate went into solution even up to the 2 gram quantity, and only a cloud was noted. The meniscus of the liquid in the flasks containing the added lead subacetate was above the 100 mark in each case, showing an increase in volume.

Experiments were tried to determine this increase in volume. Five accurately graduated flasks with glass stoppers were used, and into these were weighed the varying quantities of dry subacetate, as in the previous experiment. A 100 cc pipette was used and an equal amount of solution of sucrose was added to each flask, the flasks being shaken during the addition of the solution. When added, the flasks were corked up and allowed to stand over night. The height of the liquid being marked on the neck of the flasks, the contents were poured out and the flasks cleaned and dried. By means of a Morse-Blalock pipette, capable of reading to 0.005 cc, the flasks were filled to the mark. The results are tabulated below:

Volumes of solution of pure sucrose when adding various amounts of dry subacetate.

No.	Dry sub- acetate of lead added.	Content.	Calculated to 100.
	<i>Grams.</i>	<i>cc.</i>	<i>cc.</i>
1	0.0	99.90	100.00
2	.5	99.91	100.01
3	1.0	100.02	100.12
4	1.5	100.18	100.28
5	2.0	100.21	100.32

An increase of 0.32 cc in volume by the addition of 2 grams of dry subacetate is noted, and, with a solution polarizing 81.25° , as in the experiment given above, the calculated polarization for this increase in volume would be 80.99° . The solution actually polarized 80.9° . Horne ^a gives 0.22 cc as the increase in volume on 1 gram of subacetate. Pellet has shown it to be 0.37 cc. The referee's sugar sample for this year, worked as above described, showed the following changes in volume in two experiments:

Changes in volume using official sugar samples.

No.	Dry subacetate lead added.	Experiment 1.	Experiment 2.
	<i>Grams.</i>		
1	0.0	100.00	100.00
2	.5	100.09	100.14
3	1.0	100.25	100.19
4	1.5	100.32	100.34
5	2.0	100.53	100.58

An average increase in volume of about 0.55 cc is noted, this being due to the precipitate formed and also to the fact, as shown above, of the solution of the lead subacetate.

From these experiments it is seen that clarification with dry lead introduces the same errors as with wet lead, viz, a precipitation of the reducing sugars, and also where used to excess a change in volume. The latter effect with wet lead acetate as a clarifier tends to raise the readings, while with dry lead there is a tendency to lower them. However, in using the dry subacetate of lead the errors are compensating, since the increase in volume tends to lower the reading and the precipitation of the levulose to raise it, while with wet subacetate the volume is decreased by the formation of the precipitate, hence the reading increased, and this is again increased by the precipitation of the levulose. Dry lead subacetate is a step in advance in the search for the best clarifying agent, and further experiments are in progress; but so far the perfect clarifying agent for dark-colored sugar solutions has not been found.

As to the use of hydrosulphites as a bleach for solutions to be polarized there are serious objections. When large quantities of reducing sugars are present in the sample the reading is lowered. This was pointed out at last year's meeting by the writer. The rotation of one of the sugars, dextrose, is decidedly lowered; hence the polarization is lowered if the sample contains much dextrose. This change of rotation of dextrose is due to the formation of an oxysulphonate which has a levorotation. The dissociation of the glucose (dextrose) oxysulphonate can be measured by this fact. In the experiments cited no inversion of sucrose by this substance was noted, but later literature shows numerous cases of inversion by using commercial hydrosulphite.

Where the quantity of reducing sugars is small, there is very little reduction in the polarization due to the formation of this compound, and it has this merit, that readings are not vitiated by a change in volume due to a precipitate. These compounds, hydrosulphites, while stable under most conditions, are very easily decomposed in moist air and also on long standing, and hence lose their power of decolorization. And again, their power of decolorization is limited, as they have no effect on caramel bodies (those which give the dark color to molasses) but do bleach intermediate substances, which on longer heating would yield caramel.

^a J. Amer. Chem. Soc., 1907, 29: 928.

MOLASSES SAMPLE.

Unfortunately the sample of molasses selected for the collaboration work was of such a consistency that fermentation started after shipping, and the results are not of such value as they might have been had this not occurred. The reserve sample also was found to be fermented, so that it was not possible to make check results.

The results as received from the collaborators are given here in tabular form.

Polarization of molasses with different clarifying agents, using only amount necessary for clarification.

SUBACETATE OF LEAD.

Analyst.	Amount of clarifying agent.	Direct polarization.	Corrected invert polarization.	Temperature of polarization.	Sucrose by Clerget method.
	cc.	° V.	° V.	° C.	Per cent.
A. W. Blair, Florida.....	5	42.56	-20.00	22.0	47.51
P. H. Doherty, New Orleans, La.....	8	43.00	-18.26	26.5	47.33
W. L. Howells, New Orleans, La.....	6	43.40	-15.80	23.2	45.17
W. P. Naquin, New Orleans, La.....	8	42.42	-20.59	22.6	47.90
J. H. Parkins, Richmond, Va.....	5	42.40	-17.60	26.3	46.33
F. G. Smith, New Orleans, La.....	6	43.72	-17.35	27.5	47.38
F. Zerban, New Orleans, La.....	5	41.80	-19.36	27.0	47.35
Average.....		42.82			47.29

DRY SUBACETATE OF LEAD.

Analyst.	Grams.				
A. W. Blair, Florida.....	1.0	42.00	-19.04	22.0	46.37
P. H. Doherty, New Orleans, La.....	2.0	42.54	-17.82	27.5	46.86
W. L. Howells, New Orleans, La.....		43.10	-16.60	23.2	45.56
W. P. Naquin, New Orleans, La.....	2.0	42.36	-20.06	23.5	47.68
J. H. Parkins, Richmond, Va.....		42.60	-19.80	26.5	48.26
F. G. Smith, New Orleans, La.....		44.22	-16.85	27.5	47.37
F. Zerban, New Orleans, La.....	2.0	42.00	-20.02	26.0	47.82
Average.....		42.63			47.22

NEUTRAL LEAD ACETATE.

Analyst.	cc.				
A. W. Blair, Florida.....	2	42.00	-19.84	22.0	46.97
P. H. Doherty, New Orleans, La.....	12	42.80	-17.91	27.5	47.09
W. L. Howells, New Orleans, La.....	6	42.90	-14.00	23.0	43.42
W. P. Naquin, New Orleans, La.....	10	42.04	-20.10	23.4	47.45
J. H. Parkins, Richmond, Va.....	5	42.00	-22.00	26.3	49.49
F. G. Smith, New Orleans, La.....	6	43.47	-17.45	27.5	47.26
F. Zerban, New Orleans, La.....	5	42.00	-19.58	25.0	47.31
Average.....		42.46			47.22

BASIC LEAD NITRATE.

Analyst.	cc each.				
P. H. Doherty, New Orleans, La.....	5	42.60	-18.26	27.5	47.22
W. L. Howells, New Orleans, La.....	5	44.00	-17.40	23.2	46.86
W. P. Naquin, New Orleans, La.....	5	42.46	-20.24	23.5	47.89
J. H. Parkins, Richmond, Va.....	5	43.20	-19.80	26.5	48.68
F. G. Smith, New Orleans, La.....	5	43.90	-17.50	27.5	47.62
F. Zerban, New Orleans, La.....	5	41.90	-20.24	24.0	47.56
Average.....		43.23			47.65

ALUMINA CREAM AND SODIUM HYDROSULPHITE.

Analyst.					
P. H. Doherty, New Orleans, La.....		42.00			
W. L. Howells, New Orleans, La.....		42.40	-18.20	23.2	46.26
W. P. Naquin, New Orleans, La.....		41.96	-21.31	18.6	47.45
J. H. Parkins, Richmond, Va.....		42.00	-18.48	26.5	46.73
F. G. Smith, New Orleans, La.....		42.97	-17.53	29.0	47.21
F. Zerban, New Orleans, La.....		40.60	-21.34	24.0	47.41
Average.....		41.99			47.01

α Omitted from average.

A comparison of the average direct polarizations develops the fact that the hydrosulphites, as in the case of the sugar sample, give the lowest readings, neutral lead acetate next, and then dry and wet lead subacetate, which are about the same. The polarization with Herles' solution is the highest. The low polarization, when using hydrosulphites, has been already explained. Leaving that one out and the Herles' polarization, the other three agree fairly well. The calculations for sucrose by the Clerget formula give results that agree very closely. The highest is the Herles' result. When this reagent is used, the factor is not 142.66, but 143.5, due to the fact of the presence of a nitrate, instead of an acetate salt. Using this factor, the results would be lower.

Polarization of molasses with different clarifying agents, using an excess of clarifier.

SUBACETATE OF LEAD.

Analyst.	Amount of clarifying agent.	Direct polarization.	Corrected invert polarization.	Temperature of polarization.	Sucrose by Clerget method.
	cc.	° V.	° V.	° C.	Per cent.
P. H. Doherty, New Orleans, La.	10	43.24	-18.04	27.0	47.44
W. L. Howells, New Orleans, La.	8	a 43.50	-15.12	23.2	a 44.73
W. P. Naquin, New Orleans, La.	10	42.50	-19.75	24.2	47.68
J. H. Parkins, Richmond, Va.	10	43.60	-17.60	26.3	46.55
F. G. Smith, New Orleans, La.	8	43.92	-17.33	27.5	47.52
F. Zerban, New Orleans, La.	7.5	42.00	-19.58	27.0	47.68
	10	42.30	-18.81	26.0	47.13
Average		42.94			47.33

DRY SUBACETATE OF LEAD.

	Grams.				
P. H. Doherty, New Orleans, La.	3.0	43.04	-17.60	27.5	47.04
W. P. Naquin, New Orleans, La.	3.0	42.85	-19.05	23.0	47.19
F. Zerban, New Orleans, La.	4.0	42.40	-19.36	26.0	47.63
Average		42.76			47.29

NEUTRAL LEAD ACETATE.

	cc.				
P. H. Doherty, New Orleans, La.	15	43.00	-18.26	27.5	47.52
W. P. Naquin, New Orleans, La.	15	42.16	-20.28	23.0	47.60
J. H. Parkins, Richmond, Va.	10	a 42.00	-22.00	26.3	a 49.49
F. Zerban, New Orleans, La.	7.5	42.10	-19.58	25.0	47.39
	10.0	42.10	-19.58	25.0	47.39
Average		42.34			47.48

BASIC LEAD NITRATE.

	cc each.				
P. H. Doherty, New Orleans, La.	10	43.40	-17.82	27.0	47.39
W. P. Naquin, New Orleans, La.	10	a 44.02	-20.15	23.5	a 49.02
W. L. Howells, New Orleans, La.	10	a 44.40	-15.40	23.2	a 45.64
F. G. Smith, New Orleans, La.	10	44.30	-17.47	27.5	47.92
F. Zerban, New Orleans, La.	7.5	42.50	-19.03	26.5	47.55
	10.0	42.80	-18.70	27.0	47.62
Average		43.25			47.62

a Omitted from average.

When an excess of reagent is used all the polarizations are raised, as shown in the preceding tables. In the direct polarization, clarification with an excess of dry subacetate gives the least increase in polarization, while the greatest is noted with Herles' solution. Neutral acetate shows a lower reading when used in excess. The agreement in the Clerget calculation here is closer than in the other cases.

Summing up the work, it can be said that where reducing sugar determinations follow polarizations the clarifying agent should be neutral lead acetate. But for ordinary polarization work, where the reducing sugar content is not high, either sub-acetate or neutral acetate can be used. Where the content of invert sugar is high, a double polarization is necessary to obtain the correct figure for sucrose, and then any of the clarifying agents can be used, but care should be taken not to use an excess.

There is one point to which special attention should be called, namely, the estimation of reducing sugars. In Bulletin 107, Revised, page 53, under 6, Direct Weighing of Cuprous Oxid, the weighing as cuprous oxid will give too high results if the material under examination is high in nitrogenous matter or mineral salts; xanthin bases and some other nitrogenous bodies are thrown down by the Fehling solution along with the cuprous oxid. Also some salts are precipitated, and would be weighed as cuprous oxid, thereby giving false results. This has been conclusively shown by C. A. Browne in his reports as referee on sugar for the past two years, and is borne out in all of the referee's work. In such cases the copper of the precipitate must be determined direct either as cupric oxid or, better, by some volumetric method, as Low's, where the cuprous oxid is dissolved, treated, and finally the copper estimated by titration with thiosulphate. This is a longer procedure than the weighing as red oxid, but it should be done if reliable and accurate figures are to be obtained.

RECOMMENDATIONS ON SUGAR.

It is recommended—

- (1) That the question of the influence of precipitants upon the polarization of sugars be further investigated.
- (2) That the question of methods of determining moisture or dry substance be further investigated.
- (3) That the method of determining dry substance by means of the refractometer and the table of Geerligs be adopted provisionally by the association.
- (4) That under "Methods for the Determination of Copper contained in the Precipitate of Cuprous Oxid," pages 51-53, Official Methods, Bulletin 107, Revised (6) "Direct Weighing of Cuprous Oxid," there be a limitation inserted, viz: "This method should not be used in determining reducing sugars in commercial products, as other substances are precipitated along with the cuprous oxid. In these products the copper of the cuprous oxid should be determined direct by titration as in Low's method (*ibid.*, 241) or as cupric oxid."

DETECTION OF SMALL PERCENTAGES OF COMMERCIAL GLUCOSE IN SIRUPS AND HONEY.

By A. H. BRYAN, *Referee*.

The provisional method of this association is the one described on page 70 of Bulletin 107, Revised. It calls for a polarization of the inverted solution at 87° C. A dextro-rotary reading at this temperature is said to be due to commercial glucose. And to obtain the percentage of glucose, the method divides this reading by the factor 163 and multiplies by 100.

C. A. Browne, in his report on honey, Bulletin 110, shows that normal honey naturally has a dextrorotation at 87° C. and hence the results of a determination by this method would not express the truth. The dextrorotation of a honey is due to honey dextrins. These are of a different character from those obtained by acid hydrolysis of starch, or such as occur in commercial glucose. One point of difference is the fact that honey dextrin is not colored by iodine solution, while the dextrins of glucose, except in cases of a high conversion product, are colored by iodine. By means of this test, Beck-

man's test,^a as it is called, one can distinguish between these dextrins, and hence can say whether commercial glucose has been added. Browne called attention to the importance of this test. He also gave methods for the determination of the percentage of glucose present in mixtures. In the following table are given analyses of mixtures of different amounts of glucose and honey:

Analyses of mixtures of commercial glucose and clover honey.

Mixture.		Constant direct polarization at 20° C.	Invert polarization—		Polarization difference (87°—20°).	Invert sugar—		Calculated glucose.		
Glucose.	Honey.		At 20° C.	At 87° C.		Before inversion.	After inversion.	Invert polarization at 87°+1.63.	Invert polarization at (20° C.—17.5)+1.93.	100—(corrected polarization difference)X 100÷26.7).
Perct.	Perct.	% V.	% V.	% V.	% V.	Perct.	Perct.	Perct.	Perct.	Perct.
100		+153.8	+153.34	+144.32		30.02	30.45	88.5	88.5	
50	50	+ 67.0	+ 65.67	+ 73.81	8.14	53.67	54.50	45.3	43.1	56.9
20	80	+ 15.4	+ 13.42	+ 33.00	19.58	69.00	70.35	20.2	16.0	19.2
10	90	— 2.4	— 4.84	+ 18.59	23.43	74.42	74.12	11.4	6.6	8.8
5	95	— 11.5	— 14.30	+ 11.66	25.96	75.74	77.80	7.2	1.6	3.8
3	97	— 14.2	— 16.94	+ 9.13	26.07	76.62	78.01	5.6	.29	3.7
2	98	— 16.0	— 18.70	+ 8.14	26.84	76.64	78.34	5.0	.00	1.2
1	99	— 18.2	— 20.90	+ 6.93	27.83	77.20	78.87	4.2	.00	.0
.....	100	— 19.5	— 22.11	+ 5.94	28.05	77.68	78.93	3.2	.00	.0

In the direct and invert polarizations it is noted that there is a gradual change from a "plus" polarization to a "minus" polarization, due to the increase of the glucose percentages. With reducing sugars before and after inversion there is again a large increase with the decrease of glucose. The difference in polarization of the inverted solution at 20° and 87°, as shown in column 4, increases from 8.14 to 28.05. C. A. Browne found that nearly 95 per cent of his samples of pure honey showed a difference ranging from 23 to 30 and the lowest was about 20. Taking 23 as a low figure, a mixture of 10 per cent of glucose with honey would not be considered adulterated. If, however, the natural honey had not shown such a high difference, viz, 28.05, then 10 per cent would be easily detected by this figure; but by adding up to 5 per cent this difference is not noticeable, and also the other analytical figures would not indicate the presence of glucose. It is, however, easily distinguishable when Beckman's test is applied to the honey. In fact, with the addition of as low as 1 per cent of glucose, its presence can be recognized by this test, especially if the dextrins are precipitated by alcohol and then dissolved in water, thereby concentrating them.

In the last three columns of the table the results of determining per cents of glucose by the three different methods are given. It is seen that the method proposed by Browne gives the figures closest to the actual mixture. Obviously in honey work Beckman's test should be employed in all cases, and in the hands of ordinary chemists after a few trials it will give good results.

As to the need for such a test, it is a well-known fact that where commercial invert sugar is used in a mixture of honey, also where honeys that crystallize are used, a small percentage of glucose is quite often added to prevent this crystallization. Cases are on record of such mixtures where less than 1 per cent of glucose was added. The iodine test will indicate the presence of glucose down to that amount.

Along this line the same question comes up in the examination of sirup and molasses. As is well known, glucose is added to these products in large quantities, and again in

^aZts. anal. Chem., 1896, 35:267.

other cases it is added in smaller quantities for the same purpose as in the case of honey. To be able to determine the small quantity is the problem.

In the first place, molasses or sirup from cane may show some polarization at 87° C. on the inverted solution. This polarization is generally to the right, though there are cases where it was to the left. This dextrorotation may be due to a preponderance of dextrose in the reducing sugars of the sample due to the easy decomposition of the levulose or may be due to the decomposition products formed when the raw juice is being defecated with lime, or by chance it might come from a special fermentation of the sample forming dextran. However, it can be said not to be due to dextrins. The normal polarization of sirups and molasses has been studied in samples of known purity from Louisiana and is given in the following table:

Polarization of Louisiana molasses and sirup.

MOLASSES.

Direct polarization at 20° C.	Corrected Invert polarization—		Dry substance.
	At 20° C.	At 87° C.	
° V.	° V.	° V.	Per cent.
40.8	-20.24	+2.2	80.8
24.6	-20.9	+2.2	74.8
26.0	-18.26	+3.52	76.8
42.4	-16.94	+2.42	78.2
52.4	-16.28	+2.20	69.1
55.6	-13.59	+4.18	69.6
39.6	-18.04	+2.20	80.8
39.6	-17.82	+2.20	79.0
44.0	-17.16	+2.64	72.0
42.0	-17.60	+2.42	73.8
42.4	-17.27	+3.52	76.1
41.6	-16.94	+3.96	74.0
52.4	-17.60	+3.52	76.1
26.6	-19.8	.00	78.1
50.8	-25.08	+1.10	87.5
22.6	-16.72	+3.96	84.1
41.6	-14.74	+1.10	75.0
45.6	-15.4	+2.20	78.0

SIRUP.

48.4	-17.6	+1.98	74.3
54.0	-18.7	+3.30	68.3
50.2	-12.1	+6.16	
50.4	-14.3	+1.76	
61.8	-16.5	+2.20	
Average.....		+2.65	
Maximum.....		+6.16	
Minimum.....		0.00	

It is noted that the average is +2.65, the minimum 0.00, and maximum +6.16. The sample giving +6.16 was badly fermented, hence its high figure. There remain about 100 samples to be examined, and when these are finished more definite figures can be obtained. As far as the work has progressed, about +5.5 is the maximum for the invert reading at 87° C. The iodine method spoken of in the honey work can be used here, but the sample must receive some previous treatment before applying the test. Ten grams of the sample can be diluted with a little water (if the sample be a molasses), and shaken with 95 per cent alcohol, adding a little at a time with shaking. The precipitate settles on standing. When settled, pour off the alcohol, add a little water to dissolve the precipitate, heating if necessary, and then reprecipitate with 95 per cent alcohol. Repeat a number of times. If the solution of this material in water is still dark in color, filter through charcoal, or, better, add a drop of hydrochloric acid and

reprecipitate the dextrans with alcohol. Wash the precipitate with 95 per cent alcohol, finally dissolve in water, and then test with iodine. A blank of pure water should also be treated with the same quantity of iodine solution and run with the test. A positive test of erythro-dextrin or amylo-dextrin is sufficient proof of the presence of commercial glucose.

Mr. Davidson, as chairman of the committee to present the question of the unification of terms to the International Congress of Applied Chemistry, stated that the committee would present the question according to their instructions at the meeting to be held May 29, 1909, and report the results to the association at its subsequent annual meeting.

REPORT OF COMMITTEE A ON RECOMMENDATIONS OF REFEREES.

By R. J. DAVIDSON, *Chairman*.

NITROGEN.

Two recommendations (Nos. 2 and 4, Circular 38, p. 1) made by the referee in 1907, and referred to the referee for 1908, in regard to the use of copper sulphate in the Kjeldahl and Gunning methods, were again recommended for final action and were adopted. These changes in the official methods read as follows:

(1) Bulletin 107, revised, page 6, line 4, under "(3) Determination," after the words "sulphuric acid," insert: "From 0.1 to 0.3 gram of crystallized copper sulphate may also be used in addition to the mercury or in lieu of it."

(2) Bulletin 107, revised, page 7, line 4, under "(3) Determination," after the words "sulphuric acid," insert: "From 0.1 to 0.3 gram of crystallized copper sulphate may also be added."

Recommendations (3) and (4) offered by the referee for adoption were modified as follows and proposed by the committee for further work, which latter recommendation was adopted:

(3) Bulletin 107, revised, page 8, fourth line, after the word "time" insert: "Allow the flask to stand without heat for not less than six hours or for a shorter time with shaking at regular intervals."

(4) Bulletin 107, revised, page 8, under "(3) Determination," fifth line, after the word "and," insert the same sentence as in recommendation (3). The sentence then reads: "Add 5 grams of thiosulphate and allow the flask to stand without heat for not less than six hours or for a shorter time with shaking at regular intervals; then heat the solution for five minutes," etc.

POTASH.

It is recommended—

(1) That the cobalti-nitrite method for potash be tested during the coming year. (See p. 124.)

Adopted.

(2) That there be a further trial of the method involving the use of ammonium hydroxid and ammonium oxalate in the preparation of the solution in the determination of the potash in potash salts, as compared with the present method of direct precipitation of the potash without the use of the reagents mentioned.

Adopted.

The referee stated that he had not been able to take up the extensive investigations that would be necessary in attempting to define available potash, and offered the following resolution, which was adopted by the association:

Resolved, That in view of the fact that practically the entire available time of the referee on potash is needed for the study of analytical methods, the investigation of

the question of determining what should be designated as "available potash," provided for in a resolution adopted in 1906, be undertaken by a special referee or associate referee.

PHOSPHORIC ACID.

It is recommended—

(1) That the recommendation of 1907 be repeated, namely, that the referee on phosphoric acid shall take up for report, at the next meeting of the association, methods applicable under American conditions to the official examination of basic slag phosphates.

Adopted.

(2) That the referee make a further study of methods for the preparation of neutral ammonium citrate.

Adopted.

(3) That the referee investigate the amount of wash water to be employed in the treatment of the residue from the ammonium citrate digestion.

This recommendation was amended to include a study of the manner of filtering and was so adopted.

INORGANIC PLANT CONSTITUENTS.

It is recommended—

(1) That the method for the separation of iron and aluminum offered as an official method be referred to the referee for 1909 for final recommendation. (See p. 93.)

Adopted.

(2) That further work be done on the sodium peroxid method for the determination of total sulphur in plants and plant products (Bulletin 107, p. 23).

Adopted.

SOILS.

It is recommended—

(1) That the modified J. I. Smith method for total potassium be adopted as a provisional method and be further studied. (Circular 32, p. 4.)

This recommendation was adopted in the modified form, as presented by the committee, the referee having recommended its adoption as an optional method.

(2) That the sodium peroxid fusion for total phosphorus be continued as a provisional method and be further tested. (Bulletin 105, p. 145.)

This recommendation also was adopted in the form presented by the committee, the referee having recommended the adoption of the method as official.

(3) That the magnesium nitrate method for total phosphorus be adopted as a provisional method and be further tested. (See p. 115.)

Adopted.

(4) That the Knorr method for the determination of carbonates in soils be further studied. (Wiley's Principles and Practice of Agricultural Analysis, vol. 1, ed. 1894, p. 338; ed. 1906, p. 380.)

Adopted.

CONVERSION TABLES.

Five conversion tables, submitted by W. J. Gascoyne, of Baltimore, Md., for the consideration of the association, were referred to Committee A, which recommended that the whole question of the adoption of such tables be referred to a special committee, and after some discussion the matter was referred to the standing committee on revision of methods.

REPORT OF COMMITTEE ON FERTILIZER LEGISLATION.

The report of the committee of last year could not be sent out to interested parties until Saturday, November 7, 1908. This, of course, rendered it impossible to get any definite statements respecting the tentative definitions of fertilizers and of misbranding and adulteration. A number of replies have been received, however, a few of which, representing their general tenor, are submitted.

In the circumstances, therefore, the committee begs to report that it is desirable to postpone further action in regard to this important matter until the next meeting of the association. By that time the views of state officials, manufacturers, and farmers on the tentative definitions, etc., can be received and fully digested and placed in shape for consideration. The committee therefore recommends that it be continued with this report of progress for the purpose of a further study, in view of the criticisms which may be received on the definitions which have been submitted.

H. W. WILEY.

H. B. McDONNELL.

B. B. ROSS.

[The tentative definitions referred to in the report are as follows, together with extracts from the comments on the same received at the time of the meeting.]

TENTATIVE DEFINITIONS OF FERTILIZERS AND OF MISBRANDING AND ADULTERATION.

(1) A fertilizer shall be defined as any simple, compound, or mixed material, prepared for the purpose of selling, or sold, or offered for sale, to be applied to the soil as nourishment for plants, or as a modifier of the soil in any respect in its relation to the growth of plants. The term "fertilizer material" (or ingredients) shall include every plant-food material which is utilized, or intended to be utilized, in the manufacture, preparation, or mixing of the fertilizers defined above.

(2) A fertilizer, or fertilizer material (or ingredient), shall be deemed to be adulterated—

(a) If the percentage of any of its ingredients fall materially below the professed standard under which it is sold, whether this standard appear as a label upon the package or as a guaranty in any other way by the vendor thereof.

(b) If any of the ingredients thereof have an origin other than that indicated upon the package, or guaranteed in any other way by the vendor thereof.

(c) If any of the ingredients of the fertilizer, or fertilizer material, be in a state of combination different from that indicated by the label or guaranteed by the vendor thereof.

(3) A fertilizer, or fertilizer material, shall be deemed as misbranded:

(a) If any false name or misleading statement or design or device be affixed to any package thereof or used in any way as a representation of the materials thereof by the vendor.

(b) If any false or misleading statement respecting the origin of the material be made upon the label, or any statement or guaranty of the vendor.

(c) If any false or misleading statement be made upon the label, or by the vendor, respecting the country or origin of the materials of which the fertilizer is composed.

(d) If any false or misleading statement be made on the label, or by the vendor, respecting the virtues or qualities of the fertilizer or the materials composing it.

(e) If sold under any false name or appellation, whether such name appear upon the package or label or be given to the article by the vendor thereof.

(f) If it be an imitation of or offered for sale under the name of another fertilizer or fertilizer material.

COMMENTS.

(1) Definition (1) includes land plaster, ground limestone, etc. I would hardly favor the including of such materials, as any additional cost attached to such a substance as ground limestone would operate seriously against its use. In this State we do not consider ground phosphate rock (raw rock) among the commercial fertilizers, although its sale is in no way restricted.

(2) Would not the association do well to set a limit, or at least to suggest a limit, for each of the important plant-food elements, below which a guaranteed constituent would be considered as "materially" low?—C. A. MOOERS, *Tennessee Station*.

I have no suggestion or criticism to make with regard to the "tentative definitions of fertilizers and of misbranding and adulteration." They cover the ground fully to my mind. It is impossible to so frame definitions that there will not be a chance for difference of opinion as to what constitutes "misleading statement, or design, or device," and the best a law can do, it seems to me, is to lay down the general principles and leave it to the judgment of the individual officer in charge of the inspection work to decide whether the law in special cases has been violated as to misbranding. The recommendation of the committee as to work in the future would seem to be along the lines that give most promise of carrying the matter to a successful issue.—F. W. WOLL, *Wisconsin Station*.

Owing to the brief time in which to make a reply, I have only a few suggestions to make.

Definition 1 is more comprehensive than in most of the state fertilizer acts, and is evidently framed to include not only all materials sold as fertilizer, but, as well, all amendments. I confess I have considerable doubts about the wisdom of legislation so comprehensive at this time lest it too greatly encumber purely domestic exchanges. My present inclination is to prefer rather a law that takes into account only what are commonly recognized as commercial fertilizers.

I realize that there is considerable interstate traffic in certain lime products, as amendments, and that there is a possibility that abuse may spring up in this trade, but could it not be reached specifically rather than indirectly by including all amendments. The definition proposed is so broad that a carload of sand becomes a fertilizer if the sand is to be applied to affect the condition of the soil. The same is true of a carload of coal ashes applied for purely physical effects.

I do not believe it is a wise principle to enact police legislation far beyond present needs.

In making the above statements, I realize that the definitions for adulteration and misbranding which follow definition 1 are such that the objections I have offered to the definition for the word "fertilizer" may seem to be unnecessary, but I take it that if these definitions are adopted it will be for the purpose of advocating their incorporation in future state and national legislation, and that, in such setting, they will be accompanied by other clauses specifying the fertilizer materials that must be matters of guaranty. As soon as such clauses are introduced, the embarrassments I have in mind are likely to appear.

I desire to add that, in my judgment, the association, which is not specifically an organization charged with the execution of fertilizer acts, should not undertake the formulation and recommendation of a national fertilizer law, at least, until a full and formal conference shall have been had with the fertilizer control executive officials of the several States.—WM. FREAR, *Pennsylvania Station*.

* * * I certainly think this law would be an advantage, especially to the manufacturers, as there would be uniformity in all of the States. As the case is now many of the fertilizer manufacturers are required to get out separate printed matter for many of the States. However, I do not believe the control of sale of fertilizers should go outside of the State in which sold, as I believe it can be looked after much better by men who are in close proximity to the places where fertilizers are handled. * * *—T. L. CALVERT, *Ohio Department of Agriculture*.

On most of the recommendations I am heartily in accord with the views of the committee. In section 1, which defines a fertilizer and fertilizer material, it seems to me that some specific exemptions should be made. For instance, there are on sale in this State for the purpose of soil improvement prepared lime, limestone, land plaster, and marl, and hence our law expressly exempts barnyard manure, lime, wood ashes, and plaster when sold under their respective names. From the definition prepared by the committee there would no doubt arise the question as to whether such materials should not properly be included under a fertilizer law based on this definition. This point may not be well taken. I merely offer it for your consideration.

In a under 2 the question would naturally arise as to what is meant by "materially under the guaranty," and the interpretation of this term would be left solely to the judgment of the official in charge of fertilizer control. If it is possible to do so, I believe some definite statement as to what should be considered a material deficiency in any ingredient should be made, such a statement being based on the guaranteed content. That is, if the fertilizer was guaranteed to contain a certain percentage of ammonia, available phosphoric acid, and potash, a deficiency exceeding a certain per

cent of the guaranty would be considered as indicating intent to defraud.—W. J. JONES, JR., *Indiana Station*.

* * * My main criticism, other than those which are incorporated in the reprint as emanating from me, would be as to 2a. I believe that there should be added a proviso as follows:

"*Provided*, That if there should be a sufficient excess of other ingredients over the guaranty statement to make good the commercial equivalent of the promised plant food, the material may not be deemed adulterated." You will notice that I have put the verb in the permissive rather than the mandatory form, so as to leave it in the discretion of the inspecting officer to say whether the proviso should or should not hold in a given case. I should strongly urge, however, before any goods are branded as adulterated under this act, that resampling and reanalyzing should be resorted to.—J. L. HILLS, *Vermont Station*.

We are heartily in favor of the enactment of a national fertilizer-control law, that would furnish a broad, scientific, and economical guide on this subject to state law-makers. The law should provide for actual experiments so that the relative value of plant food from all sources could be accurately shown without prejudice. The state system of fertilizer control is all wrong, for the reason that the men who frame the laws have not a sufficient knowledge of the subject. * * * The great need of both the fertilizer industry and agriculture is positive knowledge without selfish influence or opinions not founded on facts. Terms should not be misleading, and the source from which the plant food is derived should be plainly stated. To leave these questions to the officials of the various States and the fertilizer manufacturers is a case of allowing the tail to wag the dog, and will prove a very unsatisfactory guide.—AMERICAN REDUCTION COMPANY.

REPORT OF COMMITTEE ON THE REVISION OF METHODS.

By J. K. HAYWOOD, *Chairman*.

The committee on the revision of methods presents as its report Bulletin 107, Revised, which was issued in July, 1908. The committee was empowered at the last meeting to make such changes in their first revision (Bul. 107) as were necessary to coordinate the methods and eliminate obsolete procedures. Such changes, together with the correction of typographical and other errors in Bulletin 107, were made in issuing the final revision. In submitting this report, I wish to thank the members of the committee and all those who have cooperated in the work, much patient and detailed labor having been put on it.

The report was accepted and a vote of thanks passed in recognition of the thorough manner in which the committee had discharged its office.

REPORT OF COMMITTEE B ON RECOMMENDATIONS OF REFEREES.

By B. B. ROSS, *Chairman*.

MEDICINAL PLANTS AND DRUGS.

It is recommended—

(1) That the present provisional method for assaying opium be made official. (Bul. 107, Rev., p. 201.)

Adopted.

(2) That the methods included in the referee's report be made provisional.

Adopted. (These methods were made provisional in 1907, and are only slightly modified in this year's report. (See p. 129.)

(3) That the method outlined in this year's report for acetanilid mixtures be further tested, and that additional mixtures be tested by this and such other methods as may be found desirable. (See p. 100.)

Adopted.

(4) That macroscopical and microscopic methods for examining drug products be studied during the coming year.

Adopted.

(5) That microchemical methods for the identification of alkaloids in drug products be further studied.

Adopted.

(6) That other microchemical methods be tested to determine the possibility of thus identifying medicinal plant principles.

Adopted.

(7) That pharmacological methods for testing the quality of drug products be investigated.

Adopted.

(8) That two associate referees on medicinal plants and drugs be appointed for the ensuing year.

Adopted.

SUGAR.

It is recommended—

(1) That the question of the influence of precipitants upon the polarization of sugars be further investigated.

Adopted.

(2) That the question of methods of determining moisture or dry substance be further investigated, giving special attention to the method suggested by W. D. Horne and reported in the proceedings of 1907 (Bul. 116, pp. 22-23).

Adopted.

(3) That the method of determining dry substance by means of a refractometer and Geerlign's table be adopted provisionally.

Adopted.

(4) That under the method for the determination of copper contained in the precipitate of cuprous oxid, pp. 51-53, Bulletin 107, Revised, limit section (6), "Direct Weighing of Cuprous Oxid," page 53, by the following insertion: "This method should not be used in determining reducing sugars in commercial products, as other substances are precipitated along with the cuprous oxid. In these products the copper of the cuprous oxid should be determined direct by titration as in Low's method (Bul. 107, Rev., p. 241) or as cupric oxid."

Referred to the referee for 1908-9 for investigation, with the further recommendation that the term "commercial products" be more closely defined.

FOODS AND FEEDING STUFFS.

It is recommended—

That the referee for 1908-9 take up the question of acidity in cattle feeds and consider how the results obtained by current methods can be applied to agricultural problems.

Adopted.

DAIRY PRODUCTS.

It is recommended—

(1) That the following methods given in the referee's report (p. 153) for the analysis of condensed milk be adopted as official, namely: (1) Preparation of sample; (2) total solids; (3) ash; (4) protein; and (5) lactose.

These methods were referred to the referee for 1908-9 for final recommendation and action by the association as to their adoption as official.

(2) That the methods for the determination of sucrose in condensed milk by inversion with citric acid and by inversion with hydrochloric acid be investigated by the referee for the ensuing year.

Adopted.

(3) That the determination of fat in condensed milk be studied, special attention being given to solutions of less than 20 per cent concentration.

Adopted.

(4) That the New Babcock standard, proposed by E. B. Holland and referred to Committee B, be referred to the referee for 1908-9.

Adopted.

This contemplated standard ^a is as follows:

NEW BABCOCK STANDARD.

SECTION 1. The unit of graduation for all Babcock glassware shall be the true cubic centimeter (0.998877 gram of water at 4° C.).

(a) With bottles, the capacity of each per cent on the scale shall be two-tenths (0.20) cubic centimeter.

(b) With pipettes and acid measures, the delivery shall be the intent of the graduation and the graduation shall be read with the bottom of the meniscus in line with the mark.

SEC. 2. The official method for testing Babcock bottles shall be calibration with mercury (13.5471 grams of clean, dry mercury at 20° C., carefully weighed on analytical balances, to be equal to 5 per cent on the scale), the bottle being previously filled to zero with mercury.

SEC. 3. Optional methods: The mercury and cork, alcohol and burette, and alcohol and brass plunger methods may be employed for the rapid testing of Babcock bottles, but the accuracy of all questionable bottles shall be determined by the official method.

SEC. 4. The official method for testing pipettes and acid measures shall be calibration by measuring in a burette the quantity of water (at 20° C.) delivered.

SEC. 5. The limit of error: (a) For Babcock bottles, it shall be the smallest graduation on the scale, but in no case shall it exceed five-tenths (0.5) per cent, or for skim milk bottles one hundredth (0.01) per cent.

(b) For full quantity pipettes, it shall not exceed one-tenth (0.1) cubic centimeter, and for fractional pipettes five-hundredths (0.05) cubic centimeter.

(c) For acid measures, it shall not exceed two-tenths (0.2) cubic centimeter.

REPORT OF COMMITTEE ON RESOLUTIONS.

By L. L. VAN SLYKE, *Chairman*.

(1) *Resolved*, That we express to Professor Snyder our appreciation of the able and courteous manner in which he has presided over the deliberations of the convention.

(2) *Resolved*, That whereas a national bill to regulate the composition and sale of insecticides and fungicides has been recently drawn up by a committee composed of members of the Association of Economic Entomologists, manufacturers of insecticides, and agricultural chemists interested in insecticide and fungicide analysis, which bill will be presented to Congress for approval and passage; the Association of Official Agricultural Chemists does hereby express its approval of national legislation on this subject, which legislation it is believed will be of inestimable service in protecting the farming community as well as the legitimate manufacturer and in unifying state insecticide and fungicide laws.

The report of the committee was approved.

^a For further discussion of this standard, see Twentieth Annual Report of the Massachusetts Agricultural Experiment Station, January, 1908, p. 113.

**APPOINTMENT OF COMMITTEE ON THE REVISION OF METHODS
AND RECOMMENDATIONS OF REFEREES.**

Mr. BIGELOW. It seems best in appointing this permanent committee to consider first the members of the editorial committee which had charge of the revision of the methods last year, and to appoint those who have so served for a short time and the newer members for a longer period. With this in view I will appoint—

To serve one year, J. K. Haywood, F. P. Veitch, and L. M. Tolman.

To serve two years, J. P. Street, F. W. Woll, and A. L. Winton.

To serve three years, B. B. Ross, E. M. Chace, and C. D. Howard.

Mr. Haywood will serve as chairman of the whole committee, which is to be subdivided as follows:

Committee A—Messrs. Haywood (chairman), Street, and Ross.

Committee B—Messrs. Woll (chairman), Veitch, and Chace.

Committee C—Messrs. Winton (chairman), Tolman, and Howard.

Mr. W. H. Bowker spoke at some length, urging the association to adopt some method for the estimation of available potash, as had been done in the case of available phosphoric acid, calling attention to the necessity for such action in conserving valuable by-products and furthering the interests of economic agriculture. The importance of this question had been already recognized by the association by creating a special refereeship for the consideration of the question of available potash, as recommended by the chairman of Committee A.

The place and time of meeting for the convention of 1909 was referred to the executive committee.

As it appeared from the special programme prepared for Monday that nearly all of the papers were on the subject of food adulteration, it was moved and carried that the association meet as a whole, not in sections, as originally contemplated.

The association adjourned to meet at 9 o'clock on Monday.

FOURTH DAY.

MONDAY—MORNING SESSION.

The association convened at 9 o'clock for the reading of special papers in accordance with the resolution adopted in 1907. Mr. Tolman, as chairman of Section C, presided.

METHODS RELATING TO THE RATE OF DECOMPOSITION OF ORGANIC MATTER IN THE SOIL.

By JACOB G. LIPMAN.

Chemically considered, humus is a comparatively inert substance; biologically considered, it is readily susceptible to a wide range of modification. The host of bacteria, fungi, and yeasts that inhabit it find no difficulty in inducing its transformation, which, in turn, reacts on the growth of crops. In so far as the bacteria and other microorganisms of the soil find suitable conditions of moisture, temperature, aeration, and chemical constitution, the humus will decay rapidly. In so far as these conditions are unsuitable, the decomposition will be slow; and the supply of available nitrogen, and probably of phosphorus and potassium also, will be but meager. This fact was well appreciated even before the function of bacteria in the soil was recognized, as may be seen, for instance, from some experiments by Boussingault and Loewy ^a published in 1853.

The recognition of humus as an important factor in crop production ^b has led logically to the analytical study of its decomposition products and their quantitative determination. Carbon dioxid, ammonia, and nitrates seemed important among these decomposition products not merely because of their indirect or direct action as sources of plantfood, but because of their value as indicators of quantitative reactions. Students of soils tried to establish a possible relation between the productive power of soils and their content of carbon dioxid, ammonia, or nitrates. We need only mention here in passing the investigations of Boussingault and Loewy, ^c and of von Fodor, ^d as bearing on the presence of carbon dioxid in soil air; the rather careful work of Baumann ^e on the determination and presence of ammonia in soils; and the examination by Boussingault ^f of various soils for their content of nitrates.

^a Mémoire sur la Composition de l'Air Confiné dans la Terre Végétale, Ann. chimie physique, 1853 (3), 30:3.

^b Liebig, Die Chemie in ihrer Anwendung auf Agricultur und Physiologie, 9th ed., Braunschweig, 1876, p. 26.

^c Loc. cit.

^d Deutsche Vierteljahrschrift öffentl. Gesundheitspflege, 1875, 7:205-237.

^e Ueber die Bestimmung des im Boden enthaltenen Ammoniak-Stickstoffes und über die Menge des assimilirbaren Stickstoffes im unbearbeiteten Boden, Habilitations-Schrift, Berlin, 1886.

^f Agronomie, chimie agricole et physiologie, 3d ed., 2:40.

The evidence gathered by these earlier investigators shows a distinct, though not always uniform, relation between the productive power of soils and their content of carbon dioxid, of ammonia, or of nitrates. We note that the air of fertile soil usually contains more carbon dioxid than the air of unproductive soil. Similarly, the fertile soils contain, as a rule, more ammonia and more nitrate than unproductive soils. But even admitting this, it seems hardly practicable to draw definite conclusions as to the future behavior of a soil from its content of the substances in question. The amount of carbon dioxid in the soil air is an indication of oxidation changes already accomplished, but not necessarily a guide to future oxidation intensity. The organic constituents of the humus, as well as the character of the microorganisms, may have been modified to preclude rapid oxidation. In the same way the quantity of ammonia in the soil is only a measure of past performance, and a very inadequate measure at that. As a transition product ammonia may be speedily oxidized to nitrates, or it may be transformed into protein substances by plants or fungi. Hence the quantity of ammonia present at any time in cultivated soil can not even serve to indicate past intensity of ammonia formation. As to nitrates, they, too, are not stable in the soil. Like ammonia, they may be utilized by higher plants, or by bacteria, yeasts, and molds for the production of new protein compounds. They may likewise be destroyed by denitrifying bacteria, or they may be leached out of the soil by excessive rainfall. In a word then, the amounts of carbon dioxid, ammonia, and nitrates in field soil are but an incomplete measure of past performance and a very inadequate guide as to future efficiency.

The better understanding of the functions of humus, which has gradually come in the wake of bacteriological investigations, has suggested new methods for the study of organic matter and its transformation in the soil. In experiments like those of Wollny,^a or in the more recent experiments of Stoklasa and Ernest,^b the evolution of carbon dioxid from soils kept under definite experimental conditions has been employed as a measure of the activities of the soil bacteria and of the susceptibility of the humus to decay. The same purpose has been accomplished in the experiments of Russell,^c and of Darbishire and Russell,^d by measuring the absorption of oxygen instead of the evolution of carbon dioxid. These methods enable us, therefore, to study the possible future behavior of the humus compounds under given conditions. In other words, we are enabled to secure some information concerning the relative availability of the constituents in the soil humus. For instance, it was found by Stoklasa and Ernest in a comparison of several soils that the average daily production of carbon dioxid in 1,000 grams of soil ranged from 17.5 to nearly 60 milligrams. More carbon dioxid was produced by the soil than by the subsoil, the aerobic activities being more prominent in the former, the anaerobic activities in the latter. Similarly, at 35° C. about twice as much carbon dioxid was produced as at 20° C. Darbishire and Russell found that in a number of untreated soils examined the absorption of oxygen in nine days ranged from 6 to 27 millimeters.

Analogous attempts at measuring the rate of decay of soil humus and of other organic materials have been made, not by determining the oxidation products of the carbon but of the nitrogen in the soil humus. It was well known that ammonia almost invariably appears as one of the products in the oxidation of nitrogenous materials of organic origin. It was likewise recognized after the convincing experiments of Müntz and Coudon^e that ammonia formation in the soil is a biological

^a J. Landwirtsch., 1886, 34:222.

^b Centrbl. Bakt. Para., 1905, pt. II, 14:723; also Zts. Zuckerind., Böhmen, 1907, 31:291.

^c J. Agr. Sci., 1905, 1:260.

^d Ibid., 1907, 2:305.

^e Compt. rend. acad. sci. Paris, 116:395.

process and distinct from nitrification proper. Further information was supplied by Marchal ^a in his demonstration of the intense oxidizing activities of *B. mycoides* involving the formation of carbon dioxide and of ammonia. It was perceived at the same time that the quantitative estimation of ammonia in the soil could lead to no definite conclusion because of the further changes which ammonia undergoes in the soil. The same may be said also of the quantitative estimation of nitrites.

On the other hand, the determination of nitrates in soils kept under definite conditions promised to give valuable information not only as regards the rate of decomposition of the soil humus, but also as regards the availability of various nitrogenous fertilizers. It is not surprising, therefore, to find in agricultural literature a vast amount of data bearing on the formation and accumulation of nitrates in the soil.^b We owe to these investigations a broader point of view and a deeper insight into conditions of soil, climate, and cropping in so far as they affect the oxidation of organic matter in the soil.

The many interesting facts brought to light by various nitrification experiments served to emphasize, among other things, the necessity of distinguishing the individual factors more or less prominent in the formation of nitrates. It seemed evident that, apart from conditions of moisture and temperature, the process of nitrification is directly affected by at least three important factors, viz, the physical and chemical composition of the inorganic constituents of the soil; the physical and chemical composition of the organic constituents of the soil; the character of the nitrifying, and perhaps of other bacteria present in the soil. Without going far afield, we may note in this connection the interesting experiments of Withers and Fraps.^c

We find, in the first place, decided differences in the rate of oxidation of substances like dried blood, cotton-seed meal, dried fish, tankage, bat guano, bone, and ammonium sulphate. Not only were these differences maintained, but they were in more or less close agreement with the corresponding differences brought out by digestion tests and vegetation experiments. We find also that the same nitrogenous substances were nitrified to a very unequal extent in different soils.^d For instance, in five different soils the proportions of nitrate nitrogen formed from cotton-seed meal under the conditions of the experiment were 4.4, 17.6, 22.9, 41.2, and 54.8 per cent, respectively. Evidently there were wide divergences in the physical, chemical, and bacteriological make-up of these soils.

But, interesting as are the facts just noted, we encounter in the work of Withers and Fraps ^e a fact which is even more significant in its bearing on the physiology of nitrification, namely, that in different soils ammonium sulphate and cotton-seed meal are not nitrified in the same order. The authors are therefore led to conclude that there may exist in the soil an organism or organisms capable of oxidizing organic matter (they should have said ammonia) directly to nitrites or nitrates. This assumption has been strengthened since by the investigations of Kaserer,^f who believes he has found an organism capable of changing ammonia directly into nitrate.

It would hardly be safe to theorize too much with these meager facts as a basis, but for our purpose we may accept them at their face value in so far as they tend to show the need of differentiation in the study of decay processes. The nitrates present in the soil at any time may be but a small fraction of the total amount ac-

^a Bul. soc. belge microsc., 1893, p. 83.

^b U. S. Dept. Agr., Office of Experiment Stations, Bul. 194, p. 57.

^c North Carolina Agr. Exp. Sta., Bul. 176, p. 19.

^d North Carolina Agr. Exp. Sta., Report of the Chemist, 1902-3, p. 6.

^e North Carolina Agr. Exp. Sta., Annual Report, 1901-2, p. 37.

^f Centrbl. Bakt. Para., 1906, 16 [2] : 681.

tually produced. It is well known that processes are constantly at work in the soil unfavorable to the accumulation of nitrates. Entirely apart from possible losses by leaching, there is the more or less remote but still real danger of denitrification. In addition to this there is the constant draft on the store of soil nitrates by bacteria, molds, yeasts, and algæ, not to mention higher plants when these are included in the experiment.

In view of these facts the more recent investigations on the decay of organic matter in the soil frequently attempt at least a partial differentiation of the single stages of the process. I am not aware of systematic attempts to determine albumoses and peptones among the fragments of protein decomposition in the soil. There are, however, systematic studies of ammonia formation as something independent of nitrite or nitrate formation. Indeed, we have come to accept the term ammonification (or ammonization) as expressing a definite change or series of changes. However, before taking up the discussion of methods relating to the study of ammonification, nitrification, and denitrification in the soil itself, it would be proper to consider here certain methods ^a which deal with the same reactions from a somewhat different standpoint.

The methods in question are based on the changes which occur in solutions of known composition when inoculated with a given weight of soil. For instance, a sterile solution of peptone or of gelatin, when inoculated with soil, will undergo decay, and a portion of the organic nitrogen will be split off as ammonia, which can be readily distilled off and estimated. Now, it happens that in equal quantities of the same solution inoculated with equal weights of different soils the amounts of ammonia produced may differ widely. Otherwise stated, soils vary in their ammonifying power. But since ammonification is a biological process, we are forced to the conclusion that the differences noted are due either to the unequal numbers of bacteria introduced into the sterile solutions by the different soils, or to differences in the species or vigor of the organisms, or perhaps to both. Be it as it may, the ammonification coefficients show fairly constant characteristics bearing a more or less definite relation to the productive capacity of the corresponding soils. Similarly, solutions have been prepared to favor the growth of nitrifying, denitrifying, or nitrogen-fixing bacteria looking toward the determination of the nitrifying, denitrifying, or nitrogen-fixing coefficients of soils. The latter methods have not, on the whole, proved as consistent in their results as have the ammonification methods. However, it would be out of place here to discuss them in detail, particularly since they have been considered elsewhere.^b

On the other hand, it would be well worth while to consider here the fundamental differences between the methods just outlined and those based on the study of bacteriological processes in the soil itself. We can well appreciate, of course, how 10 grams of one soil might cause the production of more ammonia in peptone solutions than 10 grams of another soil under identical experimental conditions. One soil might have two or three times as many ammonifying bacteria as another; or it might have not only larger numbers, but also species and individuals with a particularly well-developed power of ammonia formation. Moreover, it appears quite logical to assume that large numbers and vigorous species may produce large quantities of ammonia in the soil itself as well as in the culture solutions. Hence the analogy between the changes in suitable culture solutions and the returns from pot or field experiments.

Theoretically, however, this analogy could not always be expected to exist. It must be remembered that we are dealing here with micro-organisms entirely detached

^a U. S. Dept. Agr., Office of Experiment Stations, Bul. 194, p. 10.

^b New Jersey Agr. Exp. Sta., Bul. 210; Annual Report, 1905, p. 225; 1906, p. 119; 1907, p. 186.

from their normal medium, the soil. By placing a portion of the latter in an artificial culture solution we create entirely new conditions for the growth of the bacteria. Free play is given thereby to the establishment of new group relationships, and species obscure in the soil itself may come to the front. Hence it may often happen, as it often *does* happen, that the ammonification coefficients of two soils, as indicated by experiments in solution, "umsetzungsversuche," as the Germans would call them, do not at all correspond to actual conditions. We must therefore distinguish here between ammonifying *power* as referring to the numbers and species of the bacteria themselves, and what Fraps^a has recently designated as ammonifying *capacity*, as referring to the physical and chemical constitution of the soil as well as to the number and species of its bacteria.

The differentiation of the various bacteriological changes in the soil itself is not a simple matter. As already noted, the activities of the decay bacteria in the soil can not be measured by estimating the quantity of ammonia formed under normal conditions. The ammonia nitrogen does not accumulate, for reasons already noted. We can, however, create conditions in the soil precluding the further oxidation of ammonia. This may be accomplished by the addition to the soil of a sufficient quantity of dextrose or of other soluble carbohydrates, or salts of certain organic acids. The same purpose may be achieved, perhaps, by skillfully adjusting the reaction of the soil. By these means the ammonifying bacteria are permitted to grow while the nitrifying bacteria are suppressed. The ammonia accumulates in the soil and may be readily estimated.

The defect of this method lies in the fact that the formation of ammonia from the soil humus is, analytically, a comparatively slow process. A further defect is due to a probable rearrangement in the numbers and kinds of the decay bacteria, due to the materials added or the artificial conditions created. As will be seen presently, the first of these defects may be remedied without difficulty. The second can not be eliminated so easily, yet is not necessarily fatal to the successful application of the method.

Ammonia formation in the soil may be greatly intensified and the simultaneous suppression of nitrification effected in still another way. By mixing with the soil certain quantities of peptone, of urea, or of other nitrogenous organic substances we supply to the bacteria something from which ammonia may be produced readily and in comparatively large quantities. At the same time, the presence of these substances stops the growth of the nitrifying bacteria. In the practical application of this method in our laboratory we thoroughly mix 0.5 gram of peptone or 0.25 gram of urea with 100 grams of fresh soil, transfer the mixture into a beaker, adjust the moisture content by the addition of sterile water, cover the beaker with a glass dish, and place it in the incubator or closet. We usually sterilize the beaker, peptone, and urea before they are brought in contact with the soil. The latter is drawn with the customary precautions against gross contamination. At the end of three or four days the contents of the beaker are transferred to a 2-liter copper flask, about 150 cc of water added, and a sufficient quantity of magnesium oxid. The distillation and titration of the ammonia are performed in the customary manner.

Similarly, we may study nitrate formation in the soil itself by placing weighed quantities of the latter in beakers and maintaining suitable moisture and temperature conditions. At the end of four weeks (or of longer intervals, if desired) the soil is leached and the nitrites and nitrates determined in the leachings. In order to intensify the nitrification processes we may add to the soil weighed quantities of ammonium salts or of organic nitrogenous substances. The quantities of nitrate, which is the end product of various bacteriological activities, may serve to gauge the comparative rate of oxidation of the organic matter. This method may be employed—has, indeed,

^aTexas Agr. Exp. Sta., Bul. 106.

been repeatedly employed—for the study of the comparative availability of different nitrogenous substances as a source of nitrogen to plants.

Comparative studies of denitrification and nitrogen-fixation may be made by the same method. It is merely necessary to modify the cultural conditions by the addition of certain substances. In the case of denitrification, for instance, we add a known amount of potassium or sodium nitrate, leach the soil at the end of ten days, and determine the ammonia, nitrite, and nitrate nitrogen in the leachings and the total nitrogen in the residue. The initial nitrogen content of the soil being known, we have the complete data required.

The methods just outlined may be still further differentiated. We may find means to distinguish the single phases of ammonification as due to urea bacteria, spore or nonspore-forming aerobes, spore or nonspore-forming anaerobes. In the case of nitrification, we may attempt to distinguish the single phases of oxidation; in the case of denitrification the single phases of reduction; in the case of nitrogen-fixation the aerobic and anaerobic phases of the process. The applications suggested may enable us to gain an insight into the decay processes in the soil, which are imperfectly understood at present. Moreover, we shall not only gain in our ability to interpret past reactions as revealed by analysis, but also be enabled to forecast future reactions and quantitative changes of importance to plant food production and its assimilation by the growing crop.

An interesting paper on the determination of sulphurous acid and sulphites or sulphur dioxide in food products was submitted by Mr. Edward Gudeman. The paper comprised a comparison of the method adopted by the association and a modified method suggested by the author, the modification consisting in driving over the volatile products with low-pressure steam rather than by direct distillation, as in the association method. The steam is generated from distilled water and passed directly into the mass through a glass U-tube. The details of the paper are to be found in the *Journal of Industrial and Engineering Chemistry* for February, 1909.

THE POSSIBILITIES OF MUSCOVADO SUGAR AS AN ADULTERANT FOR MAPLE PRODUCTS.

By R. E. DOOLITTLE and A. F. SEEKER.

Occasionally there have been presented for entry at the port of New York shipments of a brown-colored sugar from Venezuela designated as "Melada" or "Melado." The product is generally in the form of rectangular cakes about 1 inch thick by 5 inches long by 4 inches wide. The cakes vary somewhat in color, but in general closely resemble maple sugar in appearance. Their use as an adulterant or substitute for the maple product seemed quite probable, and the finding of a large quantity of this grade of sugar in the factory of a dealer in maple products by one of our inspectors showed the necessity of making a careful examination of the product. We were surprised to find on employing the usual methods for determining the purity of maple sugar that the brown sugar gave practically the same results as does pure maple sugar. These figures, together with those of a pure maple sugar run at the same time, are given in the table:

Composition of muscovado and maple sugars.

Determination.	N.Y. 10676, light muscovado sugar.	N.Y. 10677, dark muscovado sugar.	I. S. 758-a, Vermont maple sugar.
Moisture (per cent).....	7.35	7.50	2.80
Ash (per cent).....	1.33	1.30	1.10
Polarization, direct, at room temperature (° V.).....	+80.0	+82.4	+84.0
Polarization, invert, at room temperature (° V.).....	-27.0	-26.8	-29.6
Polarization, at 86° (° V.).....	± 0.0	± 0.0	+ 0.0
Sucrose (Clerget) (per cent).....	81.4	83.1	85.6
Winton lead number.....	2.08	2.12	2.26
Malic acid value.....	1.19	1.24	

An analysis of the ash, however, showed a distinct difference, as is shown by the following data:

Analysis of the ash of muscovado and maple sugars.

Determination.	N.Y. 10676, light musco- vado sugar.	N.Y. 10677, dark musco- vado sugar.	I. S. 758-a, Vermont maple sugar.	Average of four samples of maple sugar. ^a
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Insoluble in boiling nitric acid (1:3).....	2.55	3.41	8.9	
Potassium oxid.....	50.08	49.89	23.6	26.49
Sodium oxid.....	4.85	2.32	1.6	
Calcium oxid.....	5.77	5.66	35.9	24.98
Magnesium oxid.....	2.20	2.63	3.0	
Ferric oxid.....	.29	.26	Slight trace.	
Chlorin.....	1.96	1.34	Trace.	
Sulphur trioxid.....	22.16	23.21	None.	1.82
Phosphoric acid.....	4.02	3.68	.45	
Undetermined.....	6.12	7.60	26.55	
Ratio $\frac{K_2O}{CaO} \times 100$	868	881	66	106
Ratio $\frac{SO_3}{CaO} \times 100$	384	410		7
Ratio $\frac{SO_3}{K_2O} \times 100$	44	47		7
Ratio $\frac{P_2O_5}{CaO} \times 100$	70	65	1	

^a Jones, Eighteenth Annual Report, Vermont Agr. Exp. Sta., 1905, p. 331.

The ash of the brown sugar consists mostly of potassium sulphate, while over 80 per cent of that of maple sugar is composed of carbonate of potassium and calcium, these two bases existing in approximately equal parts. From these facts one is led to believe that a determination of water-soluble and water-insoluble ash, their ratio, and their alkalinity would furnish the necessary evidence as to whether the product under examination was composed of maple sugar or muscovado sugar. As a matter of fact, these determinations when carried out on one of the samples gave the results shown in the following table:

Ash determinations and ratios of muscovado and maple sugars indicative of adulteration.

[All results reduced to a moisture-free basis.]

Determination.	N. Y. 10677, muscovado sugar.	I. S. 758-a, maple sugar.	Maple sugars. ^a
Water-soluble ash (per cent).....	1.23	0.50	0.53
Water-insoluble ash (per cent).....	.17	.64	.48
Ratio water-soluble ash water-insoluble ash	7.7	.8	1.1
Alkalinity of water-soluble ash (cc tenth-normal acid).....	.11	.49	.68
Alkalinity of water-insoluble ash (cc tenth-normal acid).....	.03	1.47	1.01

^a Average of a number of analyses made by Jones, Vermont Agr. Exp. Sta. Report, 1905.

Unfortunately a sirup prepared from the muscovado sugar fermented before time could be found to make the usual determinations, and no more of the sample remained for further work. However, it seems reasonable to assume that the sirup as well as the sugar could be detected by the high ratio of insoluble ash to soluble ash, and the low alkalinity of both. As confirmatory evidence an ash analysis should be made wherein a high percentage of potassium oxid and sulphur trioxid, together with a small amount of calcium oxid, would indicate the adulteration. It would appear also as if the phosphoric-acid content gave useful information, both of the muscovado sugars possessing notable amounts and the maple sugar little. These data are also given in the table in the form of ratios, which serve better to emphasize the contrast. As there is good reason to suspect that the brown sugar under discussion is being used by manufacturers of maple products, it seems highly important that an examination of the ash should be included in all routine analyses of these goods.

Acknowledgments are due to Mr. W. A. Bender for the ash analyses here given and to Mr. A. E. Taylor for many of the other determinations.

NOTES ON THE WINTON LEAD NUMBER OF MIXTURES OF CANE AND MAPLE SIRUP.

By R. E. DOOLITTLE and A. F. SEEKER.

Among a number of samples of cane and maple sirup mixtures examined during the past year were a few which had been mixed in the presence of one of the officials of the laboratory and were known to contain 10 per cent of maple sugar. Upon analysis it was found that these sirups gave no precipitate whatever with basic lead acetate when making the lead number determinations according to Winton's method.

A sample of the same maple sugar from which the sirups had been prepared was examined at the same time and gave a lead number of 2.31, besides other results which indicated the purity of the product, and therefore the negative results obtained with the 10 per cent mixtures caused some surprise.

Upon carefully repeating the determination it was observed that a precipitate was formed when the lead subacetate solution first came in contact with the sirup, but this was later redissolved when the whole of the reagent had been added. It was judged, therefore, that so great an excess of basic acetate prevented the usual precipitation with mixtures of this strength.

A number of portions of 5 grams each were accordingly taken, diluted to 15 cc with water, and placed in test tubes. To each of these were added different amounts of the standard basic acetate solution, varying from 0.1 to 5 cc, and after thorough shaking the turbidity noted. The tubes to which the smaller amounts of reagent had been added were perfectly clear, but a turbidity appeared as the quantity approached 0.5 cc, then came a slight precipitate, which reached its maximum at 1 cc and gradually decreased again to only a slight opalescence with 5 cc. Winton's method calls for 25 cc

of reagent for 25 grams of sugar or sirup, a proportion of 1 cc per gram of substance. The maximum precipitate was in this case produced by 1 cc to 5 grams of substance. A lead number determination was accordingly made on the 10 per cent maple sirup in question with a lead subacetate solution five times weaker than that prescribed by Winton, and the figure 0.137 obtained. This solution is much too weak to be used with pure maple sirups, as it contains only about 0.8 gram of lead (figured as metal) per 100 cc, whereas 100 grams of an average maple sugar will precipitate in Winton's method over 2 grams of lead. To show that a large excess of lead reagent is necessary to produce a normal precipitate, a sirup containing 30 per cent of cane sugar and 30 per cent of maple sugar was prepared and the lead number determined, using both the Winton solution and the one diluted five times. With the weak subacetate a lead number of 0.29 was obtained, with the strong, 0.72. In the former case there was just enough basic subacetate in the 25 cc of solution added to have precipitated all of the lead if a normal precipitation had occurred, and no lead would have appeared in the filtrate. Actually the amount of lead was insufficient for maximum precipitation and the lead number was accordingly too low. On comparing the amount of lead remaining in solution with that added it was seen that the former was 62 per cent of the latter. By the regular method the excess of lead producing a normal lead number was found to be 81.7 per cent. In the case of the 10 per cent maple sirup in which no precipitation was produced by the regular solution and in which a lead number of 0.137 was obtained with the 1 in 5 dilution, it was found that the excess of lead was 82.1 per cent. As a conclusion it would appear that at least 62 per cent excess of lead is necessary for a complete precipitation, an excess of lead much greater than 80 per cent tends to prevent precipitation, and that a zero lead number obtained by the regular method does not indicate that so-called cane and maple sirups contain no maple sugar.

It has been suggested that the lead number of the mixture containing 10 per cent of maple sugar might give normal results if the solution after addition of the lead reagent were allowed to stand longer than two hours, as was done in the previous determinations. On standing for twenty-four hours the opalescence which formed on adding the lead subacetate had collected into a very slight precipitate, which was matched in the blank by one of similar proportions, though boiled distilled water had been used in all cases. After filtering in the usual way and determining the lead number zero values were obtained as before.

THE DETERMINATION OF FUSEL OIL BY ALKALINE PERMANGANATE.

By A. S. MITCHELL and C. R. SMITH.

Fusel oil consists chiefly of a mixture of normal and isopropyl, normal and isobutyl, active amyl and isoamyl, and hexyl alcohols. The Allen-Marquardt method is in reality an estimation in terms of amyl alcohol of the higher alcohols which are dissolved and retained by carbon tetrachlorid, under fixed conditions, and converted into volatile acids by oxidation with a chromic acid mixture.

This paper is the result of an effort to learn the conditions necessary to produce definite oxidation of the various alcohols by alkaline potassium permanganate solution. It was hoped to avoid prolonged digestion with the oxidizing agent and, later, the subsequent distillation with the attendant concentration of the oxidizing mixture. Ordinary amyl alcohol, which consists of iso and active amyl alcohols, was first experimented upon. In the first effort the manganese dioxid and unreduced permanganate was not removed after acidifying for the distillation of the free acids. The mixture bumped so badly that the distillation could not be completed. It became necessary to remove the manganese dioxid and permanganic acid. Oxalic acid was tried and rejected. Hydrogen peroxid was finally selected for this purpose.

In the experiments recorded in the following table the amyl alcohol used had been dried and fractionated at boiling points between 128° and 132° C. After the oxidation 50 cc of sulphuric acid (1:4) was added and then an excess of hydrogen peroxid, and the mixture was boiled under a reflux condenser for fifteen minutes to remove any carbon dioxid. The mixture was then distilled until bumping occurred due to the separation of salts from the solution; 80 cc of water were added and distillation repeated to the same point. The valeric acids were then titrated with tenth-normal sodium hydroxid with the following results:

Amyl alcohol estimated under varying conditions, using alkaline potassium permanganate.

Time of oxidation.	Total dilution during oxidation.	Temperature of oxidat'on.	Potassium hy-droxid.	Potassium per-manganate.	Amount of amyl alcohol.		
					Quantity used.	Quantity found.	Per cent found.
<i>Minutes.</i>	<i>cc.</i>		<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	
30	60	Room.....	5	3	0.1170	0.1047	89.5
10	60	Boiling-water bath.....	5	3	.1347	.0959	71.2
10	60	do.....	5	3	.1190	.0833	70.0
20	60	Boiled, free flame.....	5	3	.1245	.0471	37.8
20	160	Room.....	5	2.5	.1080	.0960	88.9
20	160	Boiling-water bath.....	5	2.5	.1181	.0915	77.5
a 20	260	Room.....	5	2.5	.1259	.1093	86.8
10	160	do.....	5	2.5	.1161	.1040	89.6
10	160	do.....	5	2.5	.1090	.0903	82.9
30	160	0°.....	5	2.5	.1099	.1091	99.2
30	160	0°.....	5	2.5	.1156	.1126	97.4
30	160	0°.....	5	2.5	.1545	.1522	98.5
30	160	0°.....	5	2.5	.1631	.1566	95.4
30	160	0°.....	5	2.5	.1966	.1909	96.1

a 11 Hours.

At a temperature of 0° C. the oxidation of amyl alcohol to valeric acid appears to be quantitative and at higher temperatures the yield of valeric acid is decreased. Variations in time within the above limits have little influence upon the reaction. When the oxidation is too vigorous destruction occurs and the results are low. If the reaction is controlled by decreasing the temperature the oxidation is arrested at the production of the corresponding acids. The acids when produced are not easily altered and may be allowed to stand with the oxidizing agent at room temperature without appreciable change.

THE OXIDATION OF NORMAL PROPYL AND ISOBUTYL ALCOHOLS.

Similar experiments were made upon normal propyl and isobutyl alcohols, following the same procedure as in the case of amyl alcohol, and with the following results.

Estimation of propyl and isobutyl alcohols, using alkaline potassium permanganate.

Alcohol.	Amount used.	Amount recovered.	
		Quantity found.	Percentage found.
	<i>Grams.</i>	<i>Grams.</i>	
Propyl.....	0.1151	0.1156	100.4
Do.....	.1151	.1190	103.4
Isobutyl.....	.1005	.0981	97.6
Do.....	.1005	.1039	103.4

THE EXTRACTION OF PROPYL, ISOBUTYL, AND AMYL ALCOHOLS FROM ETHYL ALCOHOL SOLUTIONS.

A few trials were made upon the extraction of these alcohols by carbon tetrachlorid. In each case 100 cc of 43 per cent by volume alcohol, containing known amounts of one of the alcohols, was diluted to 115 cc, salted, and saturated sodium chlorid solution added to bring the specific gravity to 1.10, extracted with four successive portions of 40, 30, 20, and 10 cc, respectively. The carbon tetrachlorid was washed with three portions of saturated salt solution 50 cc each, and with one portion of 50 cc of saturated sodium sulphate solution. The alkali was added to the carbon tetrachlorid solution contained in a separatory funnel and cooled in an ice bath to 0° C. The permanganate, previously cooled, was added and allowed to remain in the bath thirty minutes, with repeated shaking. The distillation was conducted as previously described.

Extraction of alcohols, using carbon tetrachlorid.

Alcohol.	Quantity used.	Quantity found.	Percentage found.
	<i>Grams.</i>	<i>Grams.</i>	
Propyl.....	0.1292	0.0225	17.5
Do.....	.1292	.0237	18.3
Isobutyl.....	.1620	.0920	56.7
Do.....	.1620	.0953	58.8
Amyl.....	.2025	.1747	86.2

Attempts to extract amyl alcohol from ethyl alcohol solutions, using petroleum ether as a solvent, failed.

DIRECT VOLUMETRIC METHOD FOR THE DETERMINATION OF PROPYL, ISOBUTYL, AND AMYL ALCOHOLS.

Repeated attempts have been made during the course of this work to estimate propyl, isobutyl, and amyl alcohols, depending on the amount of permanganate reduced, but without success until satisfactory conditions had been determined for the oxidation. The permanganate consumed was greatly in excess of the theoretical amount. The unused permanganate was determined by adding a known amount of oxalic acid in excess and titrating back with permanganate.

The alcohols were contained (or dissolved) in 50 cc of water, to which 10 cc of potassium hydroxid were added and the whole cooled in an ice bath. One hundred cc of an aqueous solution containing 2 grams of potassium permanganate and previously cooled in the ice bath were then added and the mixture was allowed to stand for thirty minutes. It was then removed from the bath, acidified with 50 cc of 24 per cent sulphuric acid, a known excess of oxalic acid was added; the mixture was then warmed and the excess of oxalic acid titrated back with standard permanganate. The following factors were used as calculated from the theoretical oxidation of the alcohols to their acids by potassium permanganate: One gram of permanganate equals 0.475 gram of propyl alcohol, or 0.585 gram of isobutyl alcohol, or 0.696 gram of amyl alcohol. The results follow:

Preliminary trials for the volumetric determination of alcohols.

Alcohol.	Amount used.	Amount found.	Per cent found.
	<i>Gram.</i>	<i>Gram.</i>	
Propyl.....	0.1150	0.1245	108
Do.....	.1150	.1246	108
Isobutyl.....	.1005	.1094	109
Amyl.....	.1482	.1910	130
Do.....	.1074	.1400	130

These figures seemed fair in the cases of propyl and isobutyl alcohols, but poor for amyl alcohol. They apparently indicate the complete destruction of a portion of the latter at some stage of the process. Varying the amount of alkali made no improvement.

That the results were slightly high, as in the case of the propyl and isobutyl alcohols, might be expected, for permanganic acid and manganese dioxid are produced on acidifying and are present together for several minutes during reduction by oxalic acid. The excessive destruction of permanganate with amyl alcohol points to other causes.

As oxalic acid acts very slowly in reducing the manganese dioxid, it was decided to substitute hydrogen peroxid. Sulphuric acid was added to hydrogen peroxid and the mixture resulting from the oxidization was poured slowly into it. This gives the permanganate little opportunity to react with free organic acids, as it is reduced by the hydrogen peroxid as it is acidified.

Determination of amyl alcohol, substituting hydrogen peroxid for oxalic acid.

Amyl alcohol used.	Permanganate consumed.	Amyl alcohol found.	Per cent found.
<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	
0.1276	0.1850	0.1287	100.9
.1276	.1830	.1274	99.8
.1276	.1827	.1272	99.7
.1536	.2173	.1512	97.1
.2042	.2971	.2067	101.2

A series was run in which isobutyl and propyl alcohols were used instead of amyl alcohol.

Determination of isobutyl and propyl alcohols, substituting hydrogen peroxid for oxalic acid.

Alcohol.	Amount used.	Permanganate consumed.	Amount found.	Per cent found.
	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	
Isobutyl.....	0.1005	0.1640	0.0950	95.0
Do.....	.1005	.1645	.0960	95.0
Do.....	.1407	.2240	.1300	92.4
Do.....	.1608	.2525	.1477	91.8
Propyl.....	.1150	.1625	.0770	67.0

The results indicate incompleteness of the oxidation at 0°, so the procedure was adopted of starting the oxidation at 0° and allowing the mixture to warm slowly to room temperature. The mixture was allowed to remain ten minutes in the ice bath and taken out and warmed so that it reached room temperature (about 23° C.) in twenty minutes. The experiments reported below were conducted with propyl alcohol.

Determination of propyl alcohol after warming solution to room temperature.

Propyl alcohol used.	Permanganate consumed.	Amount found.	Per cent found.
<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	
0.1215	0.2490	0.1183	97.4
.1458	.3050	.1449	99.4
.1701	.3600	.1710	100.5
.1215	.2495	.1185	97.4
.1215	.2530	.1202	98.9
.1458	.3040	.1444	99.0

That it should be necessary to warm the solution to room temperature seems inconsistent with the results obtained on propyl and isobutyl alcohols where the acids formed were distilled. The explanation probably lies in the fact that delay occurred before the destruction of the permanganate with the hydrogen peroxid. Hence it seems a necessary condition in the quantitative oxidation of mixtures containing isobutyl and amyl alcohols by alkaline permanganate, that after the oxidation of the amyl alcohol at 0° the solution be allowed to warm to room temperature.

That amyl alcohol in carbon tetrachlorid solution should be quantitatively oxidized by alkaline permanganate was quite probable, providing the alkaline permanganate could reach the alcohol. Knowing that the oxidation of the amyl alcohol is quite rapid, and desiring to shorten the time of shaking as much as possible, it was thought five minutes of continual shaking would be sufficient. The following table indicates the amount of amyl alcohol dissolved in 100 cc of carbon tetrachlorid and the amount found:

Determination of amyl alcohol in carbon tetrachlorid solution.

Amyl alcohol used.	Amount found.	Per cent found.
<i>Gram.</i>	<i>Gram.</i>	
0.1004	0.1029	102.4
.1671	.1704	101.9
.1892	.1931	102.1
.2053	.2064	100.5
.2450	.2467	100.7

The extraction of amyl alcohol from alcoholic solution was next subjected to experiment. In the analysis of distilled liquors we are dealing with 45 to 55 per cent by volume alcohol solutions which are extracted with tetrachlorid and the tetrachlorid washed with saturated solutions of sodium chlorid and sodium sulphate to remove the ethyl alcohol, estimating the higher alcohols extracted under fixed conditions. It was thought of importance to learn how thoroughly the washing of the ethyl alcohol from the tetrachlorid was accomplished by the customary method of washing three times with three 50 cc portions of saturated sodium chlorid solution, and lastly with one 50 cc portion of saturated sodium sulphate solution. The extraction was made from 100 cc of 45 per cent by volume alcohol. The temperature of the solutions was 23° C. In four experiments the following amounts (grams) of permanganate were consumed: 0.0080, 0.0114, 0.0090, 0.0065; average, 0.0087. This indicated the retention in the carbon tetrachlorid of approximately 0.0033 gram of ethyl alcohol.

Extraction of known amounts of amyl alcohol from ethyl alcoholic solution, 45 per cent by volume, were made with the following results:

Extraction of known amounts of amyl alcohol from ethyl alcohol solution (25° C.).

Amyl alcohol present.	Total permanganate consumed.	Correction for ethyl alcohol.	Permanganate consumed by amyl alcohol.	Amyl alcohol found.	Extraction.
<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Per cent.</i>
0.0987	0.1055	0.0087	0.0968	0.0673	68.2
.1480	.1648	.0087	.1561	.1100	74.3
.2468	.2725	.0087	.2638	.1826	74.0

Extraction of similar known amounts of amyl alcohol at 17.5° C., using 100 cc of a 50 per cent by volume solution of ethyl alcohol.

Amyl alcohol used.	Total permanganate consumed.	Correction for ethyl alcohol.	Permanganate consumed by amyl alcohol	Amyl alcohol found.	Extraction.
Gram.	Gram.	Gram	Gram.	Gram.	Per cent.
0.0987	0.1205	0.0087	0.1118	0.0778	78.8
.1481	.1810	.0087	.1723	.1199	81.0
.1481	.1730	.0087	.1643	.1143	77.2
.1974	.2296	.0087	.2208	.1537	77.9

For the estimation of ethyl alcohol by alkaline permanganate a solution of fusel-free alcohol containing 24.75 grams per 100 cc as determined by the pycnometer was diluted with water one hundred times. Portions of this solution were then diluted to 50 cc and estimated by the same method as used in the case of the other alcohols. The oxidation was quite rapid, and the time allowed was ten minutes. Using the factor 0.3636 for oxidation to acetic acid the following results were obtained:

Estimation of ethyl alcohol by alkaline permanganate.

Permanganate used.	Ethyl alcohol.		
	Amount present.	Amount found.	Per cent found.
Gram.	Gram.	Gram.	
0.1392	0.0495	0.0506	102.2
.1673	.0619	.0608	98.2
.2072	.0742	.0753	101.4
.2045	.0742	.0743	100.1
.2070	.0742	.0753	101.4
.2812	.0990	.1021	103.1
.3405	.1237	.1237	100.0

Similar trials were made with methyl alcohol. Methyl alcohol was purified by drying with calcium chlorid, heating with anhydrous oxalic acid to convert into dimethyl oxalate, the crystals drained and pressed dry, and finally decomposed with slight excess of dilute alkali and distilled. The specific gravity of the distillate was taken and the methyl alcohol present obtained from Dittmar's table. In the first series of experiments the oxidation was allowed twenty minutes at the temperature of the ice bath. Considering formic acid to be the end product of the oxidation, the factor would be 0.253; if the formic acid were completely oxidized to carbon dioxid the factor would be 0.1686.

Estimation of methyl alcohol by alkaline permanganate at 0° C.

Methyl alcohol present.	Permanganate consumed.	Methyl alcohol found.			
		Factor 0.253.		Factor 0.1686.	
Gram.	Gram.	Gram.	Per cent.	Gram.	Per cent.
0.0196	0.1145	0.0289	147.5	0.0193	98.5
.0392	.2150	.0544	143.9	.0362	92.4
.0589	.3130	.0791	135.0	.0527	89.5
.0981	.4115	.1041	106.1	.0693	70.8

It will be inferred that the reaction proceeded to carbon dioxide, but was not complete when large amounts of methyl alcohol were oxidized. There is no need of cooling the solutions if complete oxidation is intended, so a series of trials were made at room temperature, time of oxidation thirty minutes, and using the factor 0.1686. The results follow:

Estimation of methyl alcohol by alkaline permanganate at room temperature.

Methyl alcohol present.	Permanganate consumed.	Methyl alcohol found.	Per cent found.
Gram.	Gram.	Gram.	
0.0196	0.1160	0.0196	100.0
.0392	.2300	.0388	99.0
.0392	.2305	.0389	99.2
.0587	.3380	.0570	97.1

When warmed in a water bath at 60° for five minutes and cooled to room temperature and titrated, 0.0393 gram of methyl alcohol gave 0.0395 or 100.8 per cent of the amount present.

For the application of the alkaline permanganate method to the carbon tetrachlorid extract of the higher alcohols from distilled liquors, the following procedure is suggested:

SOLUTIONS REQUIRED.

1. A stronger solution of potassium permanganate containing approximately 20 grams to the liter.
2. A hydrogen peroxid solution of a strength slightly in excess of that of solution No. 1 (2 per cent stronger).
3. A standard permanganate solution containing 10 grams of the salt to the liter, the value of which has been accurately ascertained.
4. A solution of potassium hydroxid of 1 : 1 strength.
5. A sulphuric-acid solution containing approximately 25 per cent of acid.

METHOD OF PROCEDURE.

To the carbon tetrachlorid extract contained in the separatory funnel, add 10 cc of the potassium hydroxid solution (No. 1). Cool the mixture in ice water to approximately 0° C. Similarly cool 100 cc of the stronger solution of potassium permanganate (No. 1), accurately measured, in a flask.

To the contents of the separatory funnel add the bulk of the permanganate solution, but without rinsing the flask and retaining the residue to be added at a later stage.

Remove the mixture from the bath and shake vigorously for a period of five minutes; set aside for thirty minutes, with occasional shaking, permitting the liquid to warm to room temperature (20° to 25° C.).

Accurately measure 100 cc of hydrogen peroxid solution into a 1-liter Erlenmeyer flask. Acidulate this with 100 cc of sulphuric-acid solution. Slowly add the contents of the separatory funnel with constant shaking, keeping the acid solution constantly in excess.

Rinse the separatory funnel and flask containing the residue of permanganate with water and add to the peroxid solution.

Titrate the excess of hydrogen peroxid remaining with the standard potassium permanganate.

Run a blank determination, using the same amounts of the stronger permanganate, potassium hydrate, hydrogen peroxid, and sulphuric-acid solution and titrating the residual peroxid with the standard permanganate as before.

The difference in amount of permanganate consumed in grams times 0.696 gives the result in terms of amyl alcohol.

METHODS OF ANALYSIS OF DISTILLED SPIRITS.

By L. M. TOLMAN and W. E. HILLYER.

The methods of the association for the analysis of distilled spirits, as given in Bulletin 107, Revised, of the Bureau of Chemistry, are for the most part the best methods available; but a few modifications and some new methods have been found to be of value.

DETERMINATION OF COLORING MATTERS.

The method which has proved to be the most satisfactory in the Bureau of Chemistry for distinguishing between natural and artificial coloring matters in distilled spirits is the qualitative Marsh test. This depends on the relative solubilities of coloring matters in ethyl alcohol, amyl alcohol, and water. The addition of amyl alcohol, when in sufficient quantity, to a mixture of 50 parts of ethyl alcohol and 50 parts of water will cause a separation of the liquids into two layers, the lower layer being largely water and the upper one a mixture of ethyl alcohol, amyl alcohol, and some water. As a result of this separation, water-soluble coloring matter can be separated from alcohol-soluble coloring matter; that is to say, caramel can be separated from the natural coloring matter of whisky. Up to the present time this has been used as a qualitative test of the greatest value, but it now appears that the method can be adapted for quantitative determination, the amount of added coloring matter present in relation to the natural coloring matter being determined. The following method has been developed and found to be entirely satisfactory:

AMYL INSOLUBLE METHOD (QUANTITATIVE MARSH TEST).

Evaporate 50 cc of the whisky just to dryness on the steam bath in a porcelain evaporating dish. Add 26.3 cc of 95 per cent alcohol to dissolve the residue, and transfer to a 50 cc flask, using water and making up to volume with water. This gives a 50 per cent alcoholic solution from which to make an extraction. [It is necessary that the extraction should be made from a solution of definite alcoholic strength, as it can be readily seen that variations in the percentage of ethyl alcohol would make a decided difference in the amount of amyl alcohol to effect the proper separation.] Place 25 cc of this 50 per cent alcoholic solution in a separatory funnel, add 20 cc of the Marsh reagent, then shake lightly so as not to emulsify. (The Marsh reagent consists of 100 cc of amyl alcohol, 3 cc of sirupy phosphoric acid, and 3 cc of water; shake before using.) Allow the layers to separate; repeat this shaking and standing twice more, and after the layers have clearly separated the last time, draw off the lower or water layer containing the caramel or water-soluble coloring matter into a 25 cc cylinder and make up to volume with 50 per cent alcohol. Compare this portion in a colorimeter with the remaining 25 cc which has not been treated with the Marsh reagent, thus directly giving the percentage of color not soluble in amyl alcohol.

The following table gives the results obtained by applying this method to straight and imitation whiskies:

Amyl alcohol tests for color in whiskies.

Description of sample.	Insoluble in amyl alcohol.	Soluble in amyl alcohol.	Description of sample.	Insoluble in amyl alcohol.	Soluble in amyl alcohol.
	<i>Per cent.</i>	<i>Per cent.</i>		<i>Per cent.</i>	<i>Per cent.</i>
Straight American whiskies:			Imitation whiskies bought in market:		
Average.....	10	90	Average.....	86	14
Maximum.....	12	88	Scotch whiskies (straight):		
Minimum.....	7	93	Average.....	7	93
Straight whiskies bought in market:			Maximum.....	8	92
Average.....	9	91	Minimum.....	5	95

It will be seen from this table that in any straight American whisky 90 per cent of the coloring matter is soluble in the amyl alcohol and ethyl alcohol layer, while in an imitation whisky 14 per cent is soluble in the upper layer. This method gives a much more complete separation of the coloring matter taken from wood and from caramel than either the water-insoluble method or the ether-soluble method, and seems to be the most reliable and satisfactory test that we now have for the detection of added coloring matter and its estimation.

Further, if a whisky contains a certain amount of caramel this method will give a partial separation, and the percentage of caramel added can be approximately estimated. The present provisional method, known as the "Crampton and Simons test," for caramel depending on the insolubility of caramel in ether, is not nearly so satisfactory as the method here presented, as the separation of the coloring matters is much less complete. The ether-soluble method, as given in Bulletin 107, Revised, page 101, is cumbersome, and calls for unnecessary special apparatus. Accurate practical results have always been obtained by the following procedure:

ETHER-SOLUBLE COLOR METHOD.

Evaporate 50 cc of the sample just to dryness on the water bath; wash into a 50 cc flask with 25 cc of alcohol and dilute to mark with water. Transfer 25 cc with a pipette to a separatory funnel and add 50 cc of ether. Shake at intervals for half an hour, let settle, draw off the aqueous layer, and make up to 25 cc with water. Mix this latter and compare with the 25 cc of the solution which were not treated with ether. Express the amount of color removed on the percentage basis as ether-soluble color.

This modification simply eliminates the special Bromwell apparatus, and the method is but little used in the Bureau of Chemistry, but the change is presented as essential in applying the method. We do, however, use the method of determining the color insoluble in water, caramel, of course, being perfectly soluble and the coloring matter of whisky being practically insoluble in water. This gives a method of separation which is very satisfactory, the procedure outlined in this laboratory being as follows:

WATER-INSOLUBLE COLOR METHOD.

Evaporate 50 cc of sample just to dryness. Take up with cold water, using approximately 15 cc, and filter, washing with water until nearly 25 cc of filtrate is obtained. Add about 26.3 cc of 95 per cent alcohol, and complete the volume to the 50 cc mark by the addition of water. Mix thoroughly and compare in a colorimeter with the color of the original sample, stating results as percentage of color insoluble in water obtained by subtracting the percentage soluble color, reading from 100.

The following table compares the results obtained by the determination of water-insoluble color with the ether-soluble color on a number of straight whiskies and on spirits colored with caramel:

Comparison of water-insoluble color and ether-soluble color on different types of distilled spirits.

Description of samples.	Water-insoluble coloring matter.	Ether-soluble coloring matter.
	Per cent.	Per cent.
69 straight rye whiskies of known source and age.....	71.0	35.0
27 straight bourbon whiskies of known source and age.....	68.1	29.5
24 compound whiskies bought in open market.....	12.2	19.3
33 imitation whiskies bought in open market.....	6.7	7.3

This table shows that results obtained by the ether-soluble method do not show enough difference between the straight whiskies and those artificially colored. That is to say, in ether the difference in solubility between whisky color and caramel is

not large enough to make a satisfactory separation, while the water-insoluble method shows a much wider difference and gives the same information, but is not so valuable as the amyl alcohol test, which makes the most complete separation of the two kinds of coloring matter. In a study of the water-insoluble method it was found best to evaporate just to dryness, and, further, that the manner of evaporation did not affect the results. It also appears that the amount of sugar present as caramel does not affect the solubility of the whisky coloring matter.

DETERMINATION OF FUSEL OIL.

The determination of fusel oil or higher alcohol is undoubtedly one of the most important ones made in the analysis of distilled spirits, giving more information as to the methods of distillation in the manufacture of the spirit than any other factor. When the examination of distilled spirits was begun, an extensive investigation was made of the Roese method as given in the official methods of the Association of Official Agricultural Chemists,^a and it was found that a great many difficulties were encountered in employing the apparatus and method as there directed. This method, depending as it does upon the relative solubilities of alcohol, chloroform, and fusel oil in each other, requires that the conditions of temperature and concentration must be very carefully controlled. The first difficulty encountered was the leaking of the stopcocks in the apparatus adopted by the association, known as the Bromwell tube, and after many experiments it appeared that this could not be overcome. The chloroform solution would invariably leak through the ground-glass stoppers, so that it became necessary to return to the older form of apparatus as designed by Roese, which has no stopcock, but is extremely difficult to fill. With this form of apparatus, however, somewhat satisfactory results were obtained.

It was found also to be absolutely necessary that the apparatus be perfectly clean and free from any oily material, and in order to insure this it was heated in a sulphuric-acid-bichromate solution after almost every determination. Unless this is done drops of water will stick to the sides of the chloroform bulb and increase the volume of the chloroform and the amount of fusel oil shown. Also it is absolutely necessary that during the whole determination the solutions and apparatus should be kept exactly at 15° C., and to this end a large constant temperature bath was built deep enough so that the tubes could be immersed completely and the shaking could be carried on in the bath itself. It was found that if the tubes were removed from the bath and shaken in the air the results were entirely inaccurate, a much larger blank being obtained. This is easily explainable. If the temperature of the room is very much above that of the bath, the shaking will raise the temperature of the solution and change the relations between the solubilities of the various liquids in each other, thus yielding results of little value.

A regular procedure was adopted in regard to the shaking. The apparatus was filled according to directions and immersed in the constant-temperature bath until all the solutions had reached the same temperature. The tube was inverted in the bath and shaken vigorously 150 times, then reversed and allowed to stand in the tank until all the chloroform had settled back into the bulb, after which a reading was made.

By using this apparatus and carefully following these details fairly satisfactory results were obtained, but at the same time the Allen-Marquardt method was tested and found to be much more convenient and accurate. In a recent paper by Doctor Dudley, of Vanderbilt University, on "The comparison of results obtained by the Roese and the Allen-Marquardt methods,"^b these same difficulties and errors in the Roese method were noted, so that it seems advisable to abandon the old Roese method and direct our attention to the Allen-Marquardt method, which apparently gives much more satisfactory results.

^a Bul. 107, Revised, p. 97.

^b J. Amer. Chem. Soc., 1908, 30, 1271.

In the work of the Bureau of Chemistry for the past year or more much work has been done to perfect the Allen-Marquardt method, and it will be discussed somewhat in detail, as some of the modifications devised improve the method and have not been published, but are of great importance in obtaining accurate results.

The method as used at the present time is the same as is given in Bulletin 107, Revised, but it has been found necessary to have the proof of the sample under examination not much above 100 in order that the volume when made up to 1.12 specific gravity will not be too great for the separatory funnels used. In the analysis of high-proof spirits, therefore, 50 cc of the sample are used for analysis and 50 cc of water added, making the product approximately 100 proof.

One point which is extremely important is that the carbon tetrachlorid used must be of the highest purity. We have found that most of the C. P. carbon tetrachlorid on the market is entirely unsatisfactory for this determination until it has been purified by oxidation with bichromate and sulphuric acid, as called for in the present provisional method. In the proper control of this purification a renewal of the bichromate and sulphuric acid mixture is necessary and often makes the process a lengthy one where the carbon tetrachlorid is very impure. For this reason the following new method, devised by A. M. Breckler, is used:

Mix the crude carbon tetrachlorid with strong sulphuric acid in the proportion of 300 cc of acid to every 3,000 cc of the carbon tetrachlorid. Shake this mixture thoroughly at frequent intervals and allow to stand over night. Then run water through the mixture continuously, by means of a glass tube inserted to the bottom of the bottle and connect with the water tap, until thoroughly washed free from acid and impurities. Draw off the water or upper layer by means of a siphon, the last portions being removed as far as possible by a pipette. Add an excess of soda solution and distil the carbon tetrachlorid from it.

The advantage of this method is that a good blank can be obtained, the process of purification is decidedly shorter, and it may be adapted to cruder carbon tetrachlorid than can the present provisional method, thus allowing cheaper material to be used. A blank should always be run on each set of determinations and if this amounts in the end to more than 0.2 to 0.3 cc of tenth-normal alkali due to the carbon tetrachlorid, the reagent is not pure enough for this determination. The impurities present in some samples of bichromate also gave trouble. It is absolutely necessary in this method that reagents of all kinds shall be entirely free from organic contamination.

In the extraction portion of the method the following precautions are necessary:

(1.) A shaking machine gives more regular conditions for extraction, each shaking process being continued for a period of two minutes.

(2) It is of advantage to have perfectly saturated sodium chlorid and sodium sulphate, and for this purpose the solutions are kept standing over an excess of the salt and continually agitated by a current of air.

(3) It has been experimentally determined that a colder temperature gives a more efficient extraction.

(4) It is also necessary to take special care to remove by complete washing with sodium sulphate all the sodium chlorid from the carbon tetrachlorid extract on account of the formation of chlorin in the oxidizing process with bichromate and sulphuric acid and the danger of this chlorin interfering with the titration by bleaching the indicator. The present provisional method calls for one washing with sodium sulphate to accomplish this result, but it has been found by experiment that two are not sufficient to remove the sodium chlorid. On the other hand, it may be that more than two washings would abstract some of the higher alcohols, therefore it appears that the present directions in the provisional method should be changed from one to two washings.

(5) It is necessary in carrying on the oxidation that the boiling of the carbon tetrachlorid with the oxidizing solution should be slow and regular, and that a high condenser should be used to insure the complete condensation of all the products. Especially is this slow boiling necessary in the following modified method which depends on the estimation of the potassium bichromate reduced during the oxidation.

The Allen-Marquardt method, modified according to these suggestions, reads as follows:

MODIFIED ALLEN-MARQUARDT METHOD.

Reagents.

Solutions of sodium thiosulphate.—Two solutions of sodium thiosulphate are used, one an approximate three-fourths-normal not standardized, and the other a tenth-normal standardized against pure potassium bichromate whose value has been obtained against pure iron.

Carbon tetrachlorid.—The purification of this reagent is a fundamental necessity. (See Breckler's method, p. 209.)

Potassium iodid solution.—Dissolve 1 gram in every cubic centimeter of water taken.

Bichromate oxidizing solution.—Dissolve 200 grams of pulverized potassium bichromate in 1,800 cc of water and add 200 cc of concentrated sulphuric acid.

Determination.

Proceed with the Allen-Marquardt method for determining fusel oil, as given in Bulletin 107, page 98, to the point of adding the oxidizing mixture. Add exactly 50 cc of the oxidizing solution to the blank and the samples by means of a pipette or burette and then oxidize under a high reflux condenser for eight hours. During the oxidation, shaking the flask with a rotary motion will prevent any isolation of spots of bichromate on the flask below the carbon tetrachlorid. Decomposition from overheating is prevented by placing between the wire gauze and the flask two thicknesses of one-fourth inch asbestos board.

Remove the flask from the reflux condenser and separate the bichromate from the carbon tetrachlorid in a separating funnel. Care must be taken that in this process no bichromate is lost and that the carbon tetrachlorid is washed free from it. Make up the bichromate solution thus obtained to 500 cc.

Measure 200 cc of this solution into a liter flask. Add 50 cc of the potassium iodid solution, 50 cc of the approximately three-fourths-normal solution of sodium-thiosulphate, and then 20 cc of concentrated hydrochloric acid. Titrate the excess of bichromate with the standard tenth-normal thiosulphate solution. If a high content of fusel oil was present in the original sample, the addition of 50 cc of the three-fourths-normal thiosulphate solution may be excessive and if such is the case a smaller amount should be added and the blank titrated in the same manner.

Treat blanks containing exactly the same amount of the reagents used in running each series of commercial samples in the same way, starting them at the point where the carbon tetrachlorid is washed with sodium chlorid. The titration of this blank, to which has been added the same amount of the three-fourths-normal thiosulphate solution, gives the value of the oxidizing mixture. The difference between this value in cubic centimeters of tenth-normal thiosulphate and that obtained on the reduced oxidizing mixture of the commercial sample in each case gives the amount of bichromate used up by the oxidation of the fusel oil present. This difference is then calculated to grams of amyl alcohol using the following factor: 1 cc of tenth-normal thiosulphate equals 0.001773 gram of amyl alcohol.

The factor used is an average one obtained by three manipulators in making 60 runs on standards containing amounts of pure amyl alcohol, varying from 0.05 to 0.5 gram as follows:

Development of factor in the oxidizing process of the Allen-Marquardt method.

Analyst.	Content of amyl alcohol.	Number of determinations.	Maximum.	Minimum.	Average.
	<i>Gram.</i>				
Boyle.....	0.05+	9	0.001885	0.001652	0.001751
Do.....	.10+	8	.001885	.001637	.001785
Albrech.....	.10—	5	.001847	.001806	.001806
Palmore.....	.10+	4	.001790	.001634	.001726
Boyle.....	.15+	9	.001824	.001721	.001776
Do.....	.20+	8	.001840	.001762	.001799
Palmore.....	.20+	5	.001751	.001710	.001710
Boyle.....	.31+	4	.001754	.001719	.001736
Palmore.....	.30+	5	.001725	.001819	.001771
Boyle.....	.42+	4	.001830	.001800	.001810
Do.....	.53+	1	.001818		.001818
			.001885	.001634	.001773

The maximum and minimum figures show that the oxidizing process carried out under normal laboratory conditions is practically uniform with respect to the varying amounts of fusel oil present. It was found that the reactions taking place between the bichromate and the amyl alcohol were little understood, and that it was impossible to calculate a factor which gave anything like the actual results obtained; so that the results of the experiments given above were made, and the surprising closeness of the figures obtained by the various analysts shows that there is a definite reaction taking place; and, while this reaction has not been figured out, the writers feel entirely justified by the results in adopting this factor.

This change in the method was developed as it was found that the final distillation of the volatile acids was not satisfactory, on account of the fact that only a portion is distilled over when the present provisional method is followed. The following table of experimental data develops the conclusion just stated and shows on an average for all contents of amyl alcohol that the percentage yield is raised from 78 to 92 per cent if the washing process is continued.

Effect of continued washing on the results obtained by the Allen-Marquardt method.

Amyl alcohol present.	Present provisional method.		Amounts of amyl alcohol recovered by additional washings (expressed as cc tenth-normal alkali).				Total yield by additional washing.	
	Amounts obtained.	Percentage obtained.	First.	Second.	Third.	Fourth.	Amount.	Per cent.
Gram.	Gram.						Gram.	
0.05.....	0.034 .037 .035 .038 .049 .067	72.0						
0.100.....	.073 .067 .066 .108	68.0						
0.150.....	.107 .111 .119	71.0						
0.200.....	.128 .123 .136 .150	63.5						
0.250.....	.146 .162 .145 .179	60.0						
0.300.....	.190 .184 .175	60.7						
0.100.....	.085	85.0	0.7	0.2	0.3	0.5	0.096	96
0.100.....	.089	89.0	.9	.3	.0	.0	.096	96
0.100.....	.096	96.0	.3	.3	.2	.0	.103	103
0.100.....	.087	87.0	.8	.3	.2	.0	.096	96
0.100.....	.090	90.0	.2	.3	.3	.1	.098	98
0.100.....	.087	87.0	.5	.4	.1	.1	.097	97
0.100.....	.074	74.0	.8	.6	.8	.4	.097	97
0.100.....	.070	70.0	2.1	.9	.3	.0	.099	99
0.200.....	.190	95.0	.5	.1	.1	.0	.196	98
0.300.....	.237	79.0	.5	.2	.2	.1	.246	82
0.300.....	.239	80.0	.4	.4	.2	.2	.250	83
0.300.....	.238	79.0	.4	.1	.2	.2	.246	82
0.300.....	.225	75.0	.8	.3	.2	.3	.245	80
0.350.....	.294	84.0	1.1	.6	.3	.2	.313	89
0.350.....	.286	81.0	.4	.8	.2	a.2	.303	87
0.350.....	.272	78.0	.5	.3	.2	.3	.283	81
Average.....		78.0						92

a By the fifth washing 0.3 cc were obtained.

The curves plotted in fig. 5 show the relationship between the percentage yields of the present provisional method, of the impractical prolonged washing method, and of the proposed modified Allen-Marquardt method. Only the figures obtained in testing the portion of the Allen-Marquardt method which follows the beginning of the oxidation process are represented.

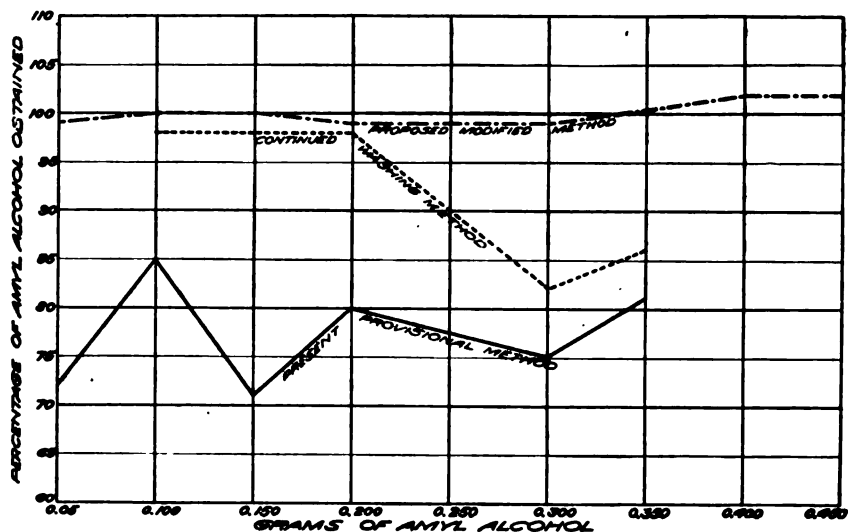


FIG. 5.—Comparison of three methods for the determination of amyl alcohol.

The curve shows that uniform results can not be obtained by the present provisional method, and that if the washing and distillation method, an impractical procedure, be adopted, high yields will probably only be obtained on the low-content samples. The curve developed by the runs on varying amounts of amyl alcohol by the proposed modification of the Allen-Marquardt method gives a higher and more uniform yield for all amounts and one which approximates closely to 100 per cent. The work of developing this latter curve represents 62 runs on varying amounts of amyl alcohol from 0.05 to 0.53 gram, and the manipulation of three analysts.

The fact of obtaining a higher and more uniform yield caused us to prepare and send out to 18 collaborators, 11 of whom reported, the samples described in the report of the associate referee on distilled spirits for this year (see p. 25). The comparison of the old and the modified method and the results of the collaborative work are there presented by the table and curves, and distinctly show the advantages of the proposed modified method.

DETERMINATION OF THE IODIN NUMBER OF THE NONVOLATILE ETHER EXTRACT OF PAPRIKA.

By W. DENIS.

As in this laboratory much difficulty was experienced in obtaining concordant results on determinations of the iodine number of the nonvolatile ether extract of paprika, 8 samples of ground paprika sent in as suspected of being adulterated with olive oil, and 1 sample composed of the shells of Hungarian paprika ground in the laboratory were examined, various methods of extraction being used.

Method 1.—This consisted in digesting 10 grams of paprika over night with 100 cc of ordinary ether in a stoppered flask. The next morning all ether was decanted off through a double filter and the residue thoroughly washed with 200 cc more ether. The ether was then distilled off and the residue dried to constant weight at 100° C. and calculated as nonvolatile ether extract, the iodine number of the same being determined on portions of this residue by the official method, using the Hanus solution.

Method 2.—Manipulation the same as in method 1 except that petroleum ether B. P. 50–60° was substituted for sulphuric ether.

Method 3.—The official method for the determination of nonvolatile ether extract in spices as given in Bulletin 107.

Method 4.—The Doolittle-Ogden method of extraction with cold anhydrous ether.^a

All four of the above methods proved unsatisfactory. Methods 1 and 2 were at once discarded because it was found after repeated trials to be absolutely impossible by these methods to obtain portions of identical composition from one lot of ether extract, due to the fact that on standing for a few minutes at 100° in the water oven several drops of a colorless oil appeared on the sides of the container, while on standing at room temperature needle-shaped crystals, which on microscopic examination proved to be crystals of fat, were seen in the deep red residue. It was also frequently found that in spite of careful filtration minute quantities of some body difficultly soluble in chloroform were present in the extract, thus further interfering with the accuracy of the results.

By the official method also it was found difficult to obtain duplicate results, apparently due to the fact that a drying oil is present in the extract which may oxidize to varying degrees depending on slight differences in manipulation during the long period of extraction. By the Doolittle-Ogden method some difficulty, although not so much as with the three preceding methods, was experienced in obtaining good duplicate determinations of the iodine number; and in addition this method has the disadvantage of allowing only one determination of the iodine number to be made on the product of a single extraction. The following modification of the Doolittle-Ogden method was finally devised, and so far has given satisfactory results, giving duplicates on iodine number determinations agreeing to within 0.2 per cent.

Method No. 5.—Ten grams of paprika spread in a thin layer on a flat-bottomed dish are dried for two hours in a vacuum oven at 60° and 25 mm. The material is then transferred to a double filter and washed with 300 cc of cold anhydrous alcohol-free ether. After distilling off the ether the residue is taken up with fresh ether and filtered into a small tared beaker, the filter paper being carefully washed with ether to remove all trace of oil. After again evaporating off the ether the residue is dried to constant weight at 100°. After the final weighing the residue is washed with chloroform into a 100 cc flask and made up to volume with this liquid. Determinations of the iodine number are made on 10 cc portions of this solution, thus making possible several duplicate determinations on the residue obtained in a sample extraction.

^aJ. Amer. Chem. Soc. 1908, 30: 1481.

The following points are noted as having been brought out by the limited data obtained:

(1) Any method in which portions of the extract are poured off into shell vials, etc., for the determination of the iodine number, as is customary in the determination of this constant with oils and fats, should be avoided, for as before stated it is absolutely impossible by this method to obtain two portions of identical composition.

(2) Iodine numbers determined on the nonvolatile ether extract of paprika, which by the high value of this constant would appear not to be adulterated with olive oil, are found when made on the product obtained by the official method of extraction to be considerably lower than those obtained on the product produced by extraction with cold solvents, indicating perhaps the presence of drying oils; while, as is to be expected, the percentage of nonvolatile ether extract is lower. On the other hand, with commercial paprikas adulterated with olive oil the difference between the iodine numbers obtained by the two methods of extraction is not so marked. Extraction with cold petroleum ether, boiling point 50°-60° gives a nonvolatile extract about 1 per cent lower than is obtained by the use of an equal volume of sulphuric ether under identical conditions. In the following table samples Nos. 1 to 8 are commercial paprikas sent in on suspicion of adulteration with olive oil while No. 9 is a pure product prepared by grinding the shells of Hungarian paprika.

Comparison of methods for determination of iodine number and nonvolatile ether extract.

No.	Iodine number.		Nonvolatile ether extract.			
	Official method.	Method No. 5.	Official method.	Method No. 5.	Method No. 1.	Method No. 2.
			<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
1	127.1	139.0	15.8	9.64	11.99	11.07
2	121.6	127.3	17.5	12.29	13.84	12.98
3	108.0	113.6	21.3	15.14	16.76	
4	124.4	130.2	15.8	11.52	13.21	12.57
5	107.7	113.3	21.75	17.00	18.73	17.97
6	109.0	114.5	20.1	15.74	17.38	16.54
7	107.3	114.4	20.6	15.63	17.28	16.36
8		130.3		13.66		
9	127.0	139.0	5.04	2.84		

DETERMINATION OF STARCH IN COCOA PRODUCTS.

By W. L. DUBOIS.

The provisional method for the determination of starch in cocoa and cocoa products requires grinding of the sample in a mortar repeatedly with ether and pouring the solution through filter paper each time until the fat is extracted. With sweetened material the fat-free residue is then rubbed in a mortar to a paste with water and filtered on the same paper, the process being repeated until all the sugar is removed, which requires about 500 cc of water. This process is a very slow and tedious one. The manipulation of the sample in the mortar with ether both in the grinding and subsequent pouring requires extreme care to prevent loss. The filtration in many cases is very slow and with sweetened samples it often takes two days to wash with 500 cc of water. In order to overcome this objection the following procedure was tried: Four grams of the unsweetened sample or 8 grams of the sweetened goods are shaken with 100 cc of gasoline in an ordinary 8-ounce, short neck, nursing bottle until the material is completely disintegrated; the bottle is whirled in a centrifuge until the supernatant liquid is clear and the gasoline drawn off with a small tube attached to vacuum pump and the process repeated. This procedure removes practically all the

fat and prepares the sample for the next operation. In case of unsweetened material this merely consists in washing the same into a 500 cc Erlenmeyer flask with 200 cc of water and proceeding with the hydrolizing and determination of starch as directed in the provisional method. With the sweetened goods after the extraction of fat with gasoline 100 cc of water are added to the residue and the bottle shaken thoroughly and whirled in the centrifugal machine. If the speed of the machine be sufficiently high a clear water solution may be obtained, although as a rule a thin layer of chocolate will float on the top. A small pipette may be passed through this layer into the water solution and the same withdrawn from the bottom. Where such high speed can not be obtained, however, it is necessary to pass the water solution through filter paper to remove the suspended particles. The process is repeated and the residue transferred to the filter paper and washed with sufficient water to make a filtrate of 500 cc. This process requires a very much shorter time than that outlined by the provisional method.

From the table it will be seen that the extraction of fat and sugar is apparently complete, the results where comparisons were made with the provisional method being slightly higher than those obtained by that method, duplicates agreeing fairly well.

Comparison of methods for the determination of starch in cocoa products.

Sample.	Modified method.	Provisional method.	Sample.	Modified method.	Provisional method.
	<i>Per cent.</i>	<i>Per cent.</i>		<i>Per cent.</i>	<i>Per cent.</i>
Cocoa nibs.....	13.05 12.68	10.77	Bitter chocolate.....	13.33 12.79	12.42 12.32
Bitter chocolate.....	11.81 12.41	11.38	Sweet chocolate.....	8.07 8.22	7.42 7.69
Bitter chocolate.....	13.15 13.64	12.50 12.90	Sweet chocolate.....	8.51 Lost.	7.80 7.40

MONDAY—AFTERNOON SESSION.

EXAMINATION OF OYSTERS.

By W. D. BIGELOW.

There has long been a practice among those shipping oysters in the shell to place them for a day or two in a stream or fresh or brackish water. This process is commercially termed "drinking," and is practiced for the purpose of plumping the oysters. It is stated that at the beginning of the ebb tide the oysters open their shells and "drink." What really happens is that the fresher water diffuses into the oysters by osmosis and gives them a fictitious appearance of plumpness.

This practice of "drinking" oysters in the shell has been largely discontinued. Practically the same thing is accomplished, however, by soaking them for a considerable time in fresh water after their removal from the shell. As the purity of the water can be better controlled by this means, it is to be preferred to the older process of "drinking" the oyster before shucking. In either case, the plumping of the oysters is stated by shippers to be for the purpose of improving the product. This improvement, however, is entirely fictitious, the increased plumpness being due merely to the addition of water which is given off on cooking.

It is believed that the unnecessary addition of water to oysters, either directly or by means of ice, is objectionable on two grounds: First, it produces a fictitious appearance of plumpness; and, second, the weight of the oysters is increased by a substance

which does not add to their nutritive value; that is, a substance (water) is mixed with them in such a manner as to reduce their quality or strength.

When the oyster is removed from the bed by dredging or tonging, the inside of the shell contains a considerable amount of dirt and sand. In order to remove this the oysters are sometimes placed on floats at some convenient place in the salt water. Here, probably at the beginning of the ebb tide, they open their shells and "drink." Since the water in which they are placed is at the same concentration as that of the bed, however, there is no plumping or other change in their appearance except that, during this process they appear to blow the dirt and sand from the shell and if the water be clean the oyster is fairly clean. They are then taken to the oyster house, shucked, and washed to remove the slime with which they are covered. It is said by shippers that this slime will rapidly produce decomposition and must be removed before the oysters are shipped. This washing, however, should not be prolonged more than is absolutely necessary for proper cleansing.

During the last season a study was made of the oysters in various parts of the country in order to secure data, if possible, by which oysters that had been properly treated might be distinguished from those which had been treated with an excessive amount of water. Seventy samples of oysters were taken from beds in various sections of the seacoast of the United States and sent to the laboratory without any treatment whatever. Other samples from the same beds were merely washed with water for a sufficient time to remove sand and dirt; while still other samples were soaked in water for a length of time varying from one hour to twenty-four hours.

When the samples arrived at the laboratory they were examined as follows: First, the total weight was taken, then the sample was strained through a colander and the solid meats and liquors weighed separately. Fifty grams of the oysters were then placed in a beaker with 200 cc of cold water and the whole heated in such a manner that the water was brought to the boiling point in about fifteen minutes. The boiling was continued for fifteen minutes, when the water was poured off as completely as possible, the oysters were cooled five minutes and weighed. From the figure thus obtained the per cent loss on boiling was determined. It was found that by continuing the boiling for a longer period than fifteen minutes the results were not greatly increased.

Another portion of the solid meats was passed through a meat chopper and the total solids, ash, and sodium chlorid determined in the usual way. The percentages of total solids and ash and sometimes of sodium chlorid were also determined in the liquor. From the figures obtained by the examination of the solid meats and liquors, together with their respective weights, the per cent of total solids in the original sample was calculated. In the majority of cases samples of the salt water were taken from the respective oyster beds and their content of sodium chlorid determined.

SIMPLE TESTS FOR DETECTING BLEACHING IN FLOUR.

By A. L. WINTON and E. J. SHANLEY.

The Griess-Ilosvay method for determining nitrites,^a originally designed for water analysis, is generally recognized as the best means of detecting artificial bleaching in flour. Commercial unbleached flour contains no appreciable amount of nitrous acid, free or combined, while that bleached with nitrogen peroxid contains amounts increasing with the degree of bleaching. The quantitative process of determining nitrous acid, although not a tedious one, is, however, unnecessarily laborious when only qualitative results are desired. It involves the preparation of a standard nitrite solution and comparison of the intensity of the color produced in this solution with

^a Sutton: Volumetric Analysis, 9th ed., 1904, p. 449.

that produced in the water extract of the sample in question, all of which can be dispensed with when the purpose is merely to learn whether or not the flour is bleached and whether the bleaching is light, moderate, or excessive. There has also been a demand not only for a simple and rapid method for detecting bleaching in the laboratory, but also for one which flour buyers, bakers, and consumers can carry out without special training and with simple apparatus.

The tests here described are, first, a simplification of the Griess-Ilosvay method and, second, a confirmatory test based on the observations of Alway^a and others that the petroleum ether solution of unbleached flour is yellow, while that of bleached flour, if not excessively overbleached, is nearly colorless. The Griess-Ilosvay test is the more reliable, but the gasoline test, which depends on an entirely different principle, namely, the nature of the coloring matter of the fat, is useful for confirmation. A description of the tests in popular language follows:

I. GRIESS-ILOSVAY METHOD.

Place a heaping teaspoonful (10 grams) of the flour to be examined in a wide-mouthed, glass-stoppered 4-ounce bottle. Nearly fill with distilled water, or tap water free from an appreciable amount of nitrites, and add a teaspoonful (4 cc) of the test solution prepared as directed below, measured with a glass spoon. Cork the bottle and shake vigorously for a few minutes, then allow to settle for from fifteen to twenty minutes.

Under the above conditions bleached flour will impart to the liquid a color ranging from a light pink to a deep red, depending on the degree of bleaching. With unbleached flour the liquid is not colored a red tint, provided water free from nitrites is used. Always run, for comparison, a parallel test with a sample of unbleached flour, so that allowance can be made for any nitrites in the water.

Test solution.—1. Dissolve 0.5 gram of sulphanilic acid in 150 cc of dilute acetic acid (about 20 per cent). Keep well stoppered.

2. Dissolve 0.2 gram of alpha-naphthylamin hydrochlorid in 20 cc of strong acetic acid (glacial), and add 130 cc of dilute acetic acid (20 per cent). Keep well stoppered.

Mix 1 and 2 for use. The mixed reagent keeps for several weeks, and possibly much longer.

II. GASOLINE METHOD.

Place two heaping teaspoonfuls (20 grams) of the flour in a wide-mouthed, glass-stoppered 4-ounce bottle, add sufficient gasoline to nearly fill the bottle, shake, and allow to settle. If the flour is unbleached, the gasoline will become distinctly yellow; if bleached, it will remain nearly colorless. Conduct a parallel test on unbleached flour for comparison.

A MODIFICATION OF THE BAMIHLE TEST FOR DETECTING WHEAT FLOUR IN RYE FLOUR.

By A. L. WINTON.

This test depends on the presence of gluten in wheat flour and its absence in considerable amounts in rye and other flours. The original test, devised in 1852 by Bamihle,^b a Prussian customs official, consists in rubbing up a small amount of flour with water on a microscopic slide by means of a cover glass and noting under the microscope whether or not gluten strings or rolls are formed. The objections to the test in its original form are that the microscope reveals the presence of traces of gluten in pure rye flour and under the microscope it is not possible to compare at a glance the amount found in pure rye flour with that from a suspected sample.

The writer's modification of the test consists in employing a dilute solution of eosin in place of water and dispensing with the microscope entirely. The gluten greedily

^aNebraska Agr. Exp. Sta., Bul. 102.

^bPoggendorff, *Annalen Physik Chemie*, 1852, 85; 161.

absorbs the dye, and on a white background becomes very conspicuous because of its beautiful pink color. A description of the procedure follows:

Place side by side on a microscopic slide 1.5 mg of the flour and a drop of water containing 0.2 gram of eosin in 1,000 cc. Allow the slide to rest on a sheet of white paper, and carefully mix the flour with the liquid by means of a cover glass, held between the thumb and finger in such a manner that it is raised slightly above the slide, taking care that none of the flour escapes from beneath it. Finally, allow the

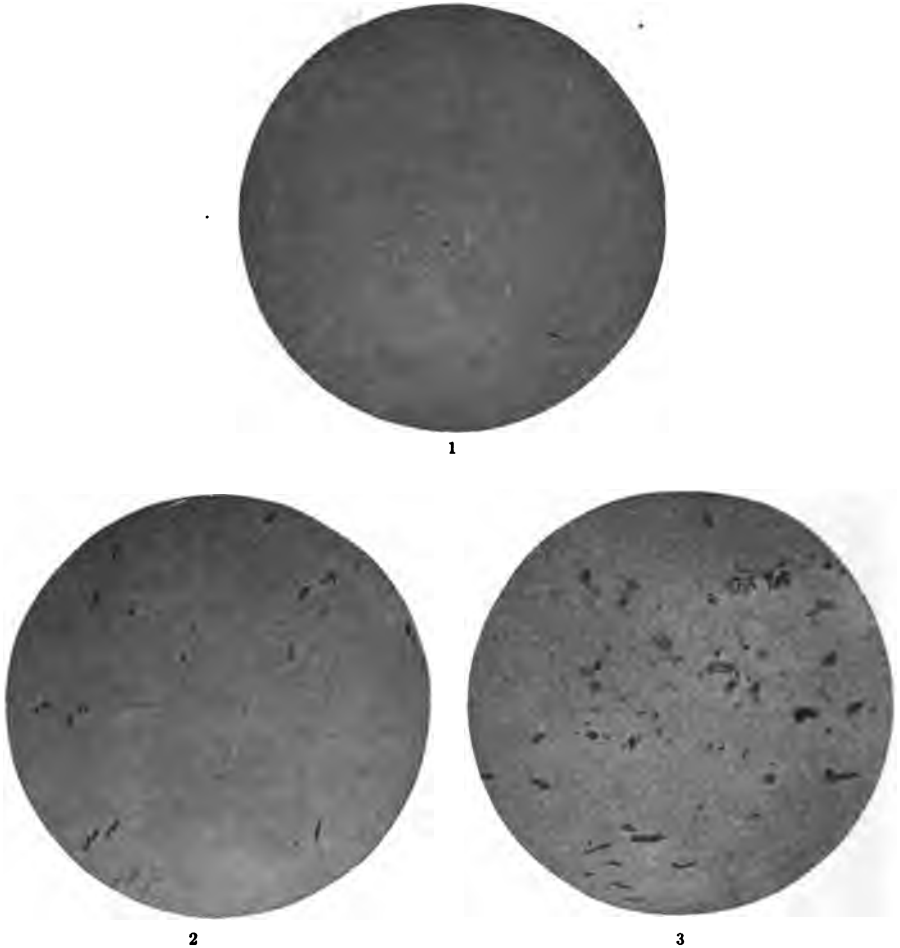


FIG. 6.—Bamhl gluten test ($\times 4$): 1, Pure rye flour showing only trace of gluten; 2, a mixture of 60 per cent rye and 40 per cent wheat; 3, pure wheat flour showing gluten masses.

cover glass to rest on the slide and rub it back and forth until the gluten, if present, forms into rolls or masses. Conduct parallel tests for comparison on pure wheat and pure rye flour. Proceeding in this manner, wheat flour yields an abundance of gluten, which is stained a beautiful pink color by the eosin, whereas rye flour yields none, or else only traces which are scarcely visible to the naked eye. Mixtures of rye and wheat flour yield variable quantities of gluten, depending upon the proportion of the two flours and their source.

In testing graham flour, buckwheat flour, and other cereal products containing considerable quantities of bran tissues or coarse lumps of any kind, the flour should be sifted through a bolting cloth before applying the test. The bolting is conveniently

carried out by placing a small quantity of flour in a beaker, covering the top with the bolting cloth held in place by means of a rubber band, inverting and shaking. Operating on the sifted material, as little as 5 per cent of wheat flour in buckwheat flour may be detected. In pure buckwheat flour I have never obtained a visible amount of gluten.

Figure 6, reproduced from photographs made by Mr. B. J. Howard, Chief of the Microchemical Laboratory, Bureau of Chemistry, shows the gluten obtained by this test in pure rye flour, a mixture of 60 per cent rye flour and 40 per cent wheat flour, and pure wheat flour, magnified 4 diameters. As has been stated, in practical work no magnification whatever is necessary to bring out the gluten strings or masses.

MOISTURE DETERMINATIONS WITHOUT THE AID OF HEAT.

By P. F. TROWBRIDGE.

For more than a year at the Missouri experiment station the moisture determinations on meats have been made without the aid of heat, by drying in vacuum over sulphuric acid. At first the ordinary brass filter pump was used for obtaining a vacuum, aided by the use of about 10 cc of ether (Benedict method). A vacuum of 1 or 2 mm is easily obtained with good water pressure, but in warm weather many of the samples would show some putrefaction before desiccation was complete enough to check decomposition. It was noted that the water formed with the acid an upper layer, which generated considerable heat when mixed by rotation. This suggested the frequent agitation of the sulphuric acid in the bottom of the desiccators, with the result that twelve hours was sufficient to dry fresh meat samples so that they would not putrefy. Having considerable difficulty with the water pressure, a Geryk duplex vacuum pump was procured, and without the aid of ether a vacuum of less than 1 mm was secured in two or three minutes.

Substances such as brain and liver gave much trouble by frothing out of the moisture tubes as the air was being exhausted, but the difficulty was obviated by freezing these samples after they were weighed. Similarly, cold-water extracts of beef were frozen and evaporated to dryness in the vacuum without ever thawing, leaving the dry substance as a web-like mass the full size of the original frozen extract.

The moisture-free samples are used for determination of the ether-soluble material. Lean meats dry down to a very hard, horn-like mass, so that it is necessary to grind them and make a second extraction in order to obtain all of the ether-soluble material. In order to avoid this difficulty, the meat samples are mixed with ignited sand, and such good results are obtained that a description of the method may be of interest.

For the moisture and fat tubes use either the S. & S. extraction shells or the glass tubes with filter-paper bottoms. Fill the tube about one-third full of ignited sea sand and then stuff in a liberal amount of fat-free cotton. Dry the tubes thus prepared (they should be numbered) for several hours in the oven at 103° C., and place in a vacuum for a few hours before weighing. Weigh in a glass-stoppered weighing bottle and record the weight of the tube and bottle consecutively. This weighing is done in advance of a slaughtering experiment, and several hundred tubes are prepared.

Place the finely ground and thoroughly mixed samples of meats in weighing bottles provided with short aluminum scoops (a heavy piece of stirring rod will do), and weigh by difference, using from 5 to 10 grams for a sample. Remove the cotton from one of the tared tubes, placing it on the side of a flat shallow porcelain dish, and carefully pour out the sand into the dish. Place the sample of the meat upon the sand and mix, using a spatula and a stirring rod. When the sand and sample are thoroughly mixed, transfer the mass to the tube, using the cotton to wipe all traces from the dish, the spatula, and the stirring rod. Loss of any particles of sand is prevented by working over black glazed paper. The last of the unused cotton is placed in the top of the tube as a plug.

Make the determinations in triplicate and place them in separate desiccators. (We use a good 6-inch vacuum desiccator. Larger desiccators were tried, but several of them broke, owing to the high pressure.) Wire-gauze baskets are used, which set on the porcelain desiccator plate. In this basket from eight to twelve tubes can be placed. The desiccator covers and stopcocks must be well ground and a lubricant

used which will hold and yet permit the easy removal of the cover. A mixture of 3 parts paraffin (hard) and 5 parts vaselin is satisfactory. These are melted together and then cooled slowly, with continual stirring. If the work is to be done during continued cold weather a little more vaselin may be used, or in summer a little more paraffin. The addition of rubber or Venice turpentine to the lubricant has been discontinued on account of the difficulty in removing the covers.

After the filled desiccators have been exhausted, rotate them carefully every three or four hours to mix thoroughly the acid and the water which has been absorbed into the upper portions. Care must be used not to spatter the acid upon the tubes. At the end of twenty-four to forty-eight hours, as is convenient, allow air to bubble slowly through concentrated sulphuric acid into the desiccator and transfer the tubes to a desiccator provided with fresh acid. Chemically pure sulphuric acid must be used, as the commercial acid discolors the samples.

Exhaust the freshly filled desiccators and hold for another twenty-four to forty-eight hours, as is convenient. During this interval mix the acid three or four times. Next weigh the tubes and place them in a vacuum again for twelve hours or longer and again weigh, to prove that the drying is complete. If any of the tubes do not show constant weight they are placed in vacuum again with fresh acid. The acid employed for the first drying is used for commercial acid; that with which the drying is completed is used as the first acid with fresh samples.

With blood the freshly drawn sample is rapidly poured into tared tubes filled with fat-free cotton, each tube being placed in a tared weighing bottle. The tube and stoppered bottle are weighed to get the weight of the sample, and the moisture is obtained as with meat samples.

We have demonstrated that this method is capable of practical application to agricultural analyses in general, and is especially to be recommended where a determination of the fat or ether-soluble constituents is to be made. The most marked differences have been noted in fat determinations upon samples of fresh bone (skeleton of beef). When heat has been used in drying the samples by the official method the extracted fats are frequently very dark colored. By using the vacuum method without heat the extracted fat is almost snow white. This method has been compared with the regular official method upon numerous other samples, as butter, milk, soil, feed stuffs, honey, soap, etc. A few results are given in the following tables illustrating several phases of the work.

Moisture determinations on various animal substances by vacuum method without heat.

Sample.	Results in triplicate.			Sample.	Results in triplicate.		
	(1)	(2)	(3)		(1)	(2)	(3)
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Blood.....	79.23	79.25	79.55	Round lean.....	72.67	73.22	72.61
Do.....	82.29	82.73	82.83	Kidney fat.....	5.52	5.51	5.42
Liver.....	68.77	68.74	68.96	Do.....	8.95	8.74	8.34
Do.....	68.82	68.82	68.41	Offal fat.....	12.51	12.29	12.43

Moisture determinations on blood by vacuum method without heat.

[Using absorbent cotton and showing effect of second drying in the vacuum.]

Weight of sample.	Weight of sample, tube, and weighing bottle.	First dry weight.	Second dry weight.	Loss in weight second time in vacuum.	Total loss in moisture.	Moisture.
<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Per cent.</i>
6.4340	46.3238	41.1025	41.0984	0.0041	5.2254	81.216
3.7002	43.3378	40.3335	40.3324	.0011	3.0054	81.223
6.3934	44.0746	38.8884	38.8860	.0024	5.1886	81.156
5.3474	44.5125	40.1695	40.1685	.0010	4.3440	81.236

Moisture determinations on a sample of liver, vacuum method without mixing with sand, showing gradual loss of moisture.

Weight of sample.	Weight of container and fresh sample.	First dry weight.	Second dry weight.	Loss during second time in vacuum.	Third dry weight.	Loss during third time in vacuum.	Fourth dry weight.	Loss during fourth time in vacuum.	Fifth dry weight.	Loss during fifth time in vacuum.	Total loss in moisture.	Moisture.
Gms.	Grams.	Grams.	Grams.	Gm.	Grams.	Gm.	Grams.	Gm.	Gms.	Gm.	Gms.	Per ct.
6.9118	45.5373	40.7115	40.6977	0.0138	40.6875	0.0102	40.6845	0.0030			4.8528	70.210
7.1317	45.4085	40.4024	40.3888	.0136	40.3850	.0038					5.0235	70.439
8.0140	46.1308	40.5963	40.5435	.0568	40.5215	.0240	40.5121	.0084	40.5070	0.0051	5.6298	70.250

Comparison of moisture determinations made by the vacuum method with those made by the official method.

Sample.	By vacuum method.		By official method.	
	Per cent.	Per cent.	Per cent.	Per cent.
Wheat stubble, air dry.....	6.565	6.525	6.975	6.945
Soil, air dry.....	2.39	2.40	1.95	1.83
Corn chop, air dry ^a	13.54	13.57	13.36	12.88
Butter ^b	16.34	16.32	16.61	16.48
Cheese.....	32.96	32.97	33.44	33.25
Milk ^b	86.68	86.62	86.71	86.76

^a The corn chop was placed in vacuum a fourth time before it ceased to lose in weight.

^b The milk and butter were mixed with sand and were dry by the vacuum method at the end of twelve hours.

THE UNIFICATION OF SACCHARIMETRIC OBSERVATIONS.

By C. A. BROWNE.

In an article upon the Control of Saccharimeters by Otto Schönrock,^a it is shown that "the differences in rotation for sugar solutions disappear for different observers and different sources of white light only when the light is filtered through a 1.5 cm column of 6 per cent potassium bichromate solution in water." On the basis of these observations Schönrock recommends that the use of this light filter be adopted in defining the 100 point of the Ventzke scale. This recommendation, which has been followed by the Imperial Reichs Anstalt of Germany and the U. S. Bureau of Standards, seems, however, to be more or less disregarded by many chemists who work with saccharimeters.

The methods of the Association of Official Agricultural Chemists say nothing as to the use of potassium bichromate solution in saccharimeters, it being deemed perhaps a necessity too well known to require mention. We know, however, of chemists purchasing saccharimeters and using them for years blissfully ignorant of the presence of the empty cell in the end of their instruments or of the purpose for which the cell was intended to be used. Their mistake, which is due usually to inability to read the German directions which accompany the instrument, is perhaps pardonable. Less pardonable is the attitude of those chemists who, knowing of the cell and the purposes of its use, yet wilfully neglect it. One very common and most fallacious argument advanced against using the cell is that standardized quartz plates polarize correctly without it and that its use is therefore wholly unnecessary. Another reason given is that the bichromate renders the polarization of dark colored solutions so difficult that it is more convenient to eliminate it altogether.

^a Zts. Ver. d. Zuckerind., 41: 521-58.

The directions of instrument makers as to the use of bichromate and the strength of solution to be employed are not at all explicit. One maker directs merely that the bichromate cell must always be filled with bichromate solution, which, however, can be more or less concentrated, according to the character of the liquid under examination.

The purpose of the bichromate solution in saccharimetric work is, of course, to correct the difference in rotation dispersion between cane sugar and quartz. The rays of light in the blue and violet which cause the greatest amount of rotation dispersion are absorbed by the bichromate. To gain more exact information as to the effect of eliminating the bichromate solution in the polarization of raw sugars I have recently compared the absorption spectra of bichromate solution with those of different clarified sugar solutions. Molasses sugars when clarified give a brownish yellow liquid, which absorbs practically all of the light in the blue and violet part of the spectrum. Solutions of such sugars act themselves as light filters and absorb the rays producing the greatest dispersion disturbances. They show upon polarization but little difference between filtered and unfiltered light. Clarified solutions of several low-grade beet sugars were found to absorb all of the violet but only a part of the blue. Slight rotation dispersion was obtained without the bichromate cell. Ninety-six degree centrifugal sugars give usually straw-colored solutions, which absorb most of the violet, but practically nothing of the blue. Rotation dispersion with these sugars is usually well marked without bichromate. Java and other high-grade sugars give upon clarification nearly colorless solutions which show very pronounced rotation dispersion without the bichromate. With such sugars the difference in reading with and without bichromate was in some cases nearly 0.2 per cent for the same observer.

The error due to rotation dispersion was found by Schönrock to be variable with different observers, a circumstance due perhaps to some physiological difference in the pigment of the eye. Comparisons which I have made on five sugars polarizing over 96° , using no bichromate and 1 and 3 per cent solutions of bichromate in a 3 cm cell, showed that the discrepancies in the readings of the same solution between four observers were augmented six and one-half times, when no bichromate was used, as compared with the 3 per cent bichromate, and two and one-half times when 1 per cent bichromate was used, as compared with the 3 per cent. Using a 3 per cent solution of bichromate in a 3 cm cell the average difference between the readings of the lowest of the four observers and the other three was only 0.03° V., using a 1 per cent solution the average difference was 0.08° V., and using no bichromate 0.22° V. The 3 per cent bichromate in a 3 cm cell gives the same effect as the 6 per cent bichromate in a 1.5 cm cell advocated by Schönrock. The use of bichromate of the above concentrations according to the length of cell should therefore be prescribed and rigidly adhered to in the polarization of sugars.

These concentrations apply, however, only to cane sugar. With substances of greater rotation dispersion such as commercial glucose, dextrin, malt products, etc., it will be found necessary to increase the strength of the bichromate considerably, as may be seen from the following:

Polarizations of starch conversion products with and without bichromate ($^\circ$ V.).

Starch conversion products.	No bichromate.	Strength of bichromate, 3 cm cell.		
		0.5 per cent.	3 per cent.	6 per cent.
Dextrin.....	253.65	253.50	253.40	253.00
Malt sirup.....	195.80	195.50	195.40	195.15
Glucose sirup.....	179.90	179.70	179.70	179.55
Do.....	172.10	171.85	171.75	171.55

With starch conversion products it is possible to secure concordant readings between different observers only when 6 per cent bichromate is used in a 3 cm cell. With substances of higher dispersion than dextrin it would seem advisable to use only sodium light for polarization. With all carbohydrate materials it would seem that the dispersion disturbances of white light may be eliminated by means of bichromate solution. The results show, however, that the directions for operating saccharimeters should specify the exact strength of bichromate solution to be used.

A second and very discordant element in the unification of saccharimetric observations is in the use of clarifying agents. The several errors resulting from the use of lead salts in clarifying sugar solutions have long been recognized. There is, first, the volume of precipitate error; second, the precipitation of levulose error; third, the formation of soluble lead levulosate of lower specific rotation than levulose; and, fourth, when dry defecation is used, the error of dilution or change in volume.

In studying these various questions my attention was directed first of all to the great difference in composition of the commercial preparations of lead subacetate and also of the solutions of this salt as ordinarily prepared for laboratory use. Preparations of the anhydrous subacetate of lead sold by reliable chemical firms, and all guaranteed as to purity according to the food and drugs act, were found to vary in their content of basic lead oxid from 3.34 to 32.32 per cent. Solutions of lead subacetate prepared by digesting litharge with the normal acetate of lead, according to the method of the association or other directions, will also vary greatly in composition, according to the time and temperature of digestion. Solutions of the same specific gravity thus prepared were found to vary in the ratio of combined to basic PbO of from 5:2 to 1:1. These variations in composition are not surprising when it is remembered that three well-defined subacetates have been prepared by the digestion of litharge with normal lead acetate. These are $3\text{PbAc}2\text{PbO}$, the subacetate ordinarily prescribed for clarification; PbAcPbO , the monobasic acetate; and $\text{PbAc}2\text{PbO}$, the dibasic acetate.

The official directions for preparing basic lead acetate are explicit as to the specific gravity of lead solutions to be used, but are silent as to the point of greatest importance, the content of basic lead. The differences which may result in saccharimetric work from the use of lead solutions of varying basicity may be seen from the following polarizations made upon a sirup and a sugar using three different solutions of lead subacetate and one solution of the normal acetate all of 1.24 specific gravity.

Comparison of polarizations using different solutions of lead subacetate.

Material	Quantity of reagent.	Normal lead acetate.	Lead subacetate.		
			$5\text{PbAc}2\text{PbO}$	$3\text{PbAc}2\text{PbO}$	PbAcPbO
	cc.	°V.	°V.	°V.	°V.
Sirup.....	8	45.00	45.50	45.85	46.00
Sugar.....	10	81.85	82.30	82.40	82.50

The solutions of greatest basicity have the greatest clarifying power and give the highest polarizations owing to the greater precipitation and lowering of polarization of the levulose and consequent increase in dextro-rotation. The 3:2 subacetate is the one usually prescribed, and since this compound can be obtained of satisfactory purity from one chemical house at least it might be well for chemists desiring uniformity to prepare these solutions directly from this salt. The important point, however, is that in whatever way prepared the solutions of basic lead used in saccharimetry should have not only a constant specific gravity, but a uniform content of basic lead.

The errors due to the volume of lead precipitate, a most serious one in the polarization of low-grade saccharine products, have been very largely eliminated by the ingen-

ious method of dry defecation proposed some years ago by W. D. Horne. The questions of change in volume and precipitation of levulose, when the dry subacetate is used in large amounts, as is always necessary with low-grade products, have given rise, however, to some uncertainties, and the method has not met with universal approval. To determine exactly the amount of error due to change in volume and precipitation of levulose, mixtures of sucrose and invert sugar, with mineral and organic salts precipitable by lead, were prepared and the effects produced upon the polarization of these solutions by different quantities of the dry acetate and dry subacetate of lead noted. It was found that in quantities up to 0.5 gram but very little change could be detected in the polarization of the original solution when either the dry acetate or dry subacetate of lead was used. Using more than 0.5 gram of substance the dry acetate invariably reduced the polarization owing to the increase in volume produced by the dissolved salt; the effect of increased quantities of the dry subacetate, however, was variable. Where but little invert sugar was present there was the same decrease in polarization owing to increase in volume. Where considerable invert sugar was present, however, this dilution error was counterbalanced, and often more than counterbalanced, by the precipitation and lowering of the specific rotation of the levulose and there was either no change in the reading of the original solution or an increase. Used in very large excess beyond the precipitation of the levulose the dry subacetate produced a continuous lowering of the polarization through dilution.

The same facts were noted in connection with the polarization of commercial sugars, as may be seen from the following polarizations made in New York by M. H. Wiley:

Effect produced by different quantities of acetate and subacetate.

Sample.	Subacetate solution.		Dry lead acetate.				Dry subacetate.			
	cc.	° V.	Gram.	° V.	Gram.	° V.	Gram.	° V.	Gram.	° V.
1. Java sugar.....	1	98.10	0.3	97.95	2.0	97.65	0.5	97.90	2.0	97.75
2. Philippine mats.....	3	88.95	1.5	88.45	3.0	88.15	1.5	88.65	3.0	88.65
3. Cuba molasses.....	1	90.75	0.5	90.60	2.0	90.40	0.5	90.65	2.0	90.55
4. Do.....	4	86.15	2.0	85.55	4.0	85.30	2.0	85.70	4.0	85.70

The dry normal acetate by the addition of excess produced dilution in every instance as is seen by the diminished polarization. This same dilution is noticed by the dry subacetate, but to a much less extent on samples 1 and 3; on samples 2 and 4 doubling the quantity of dry lead subacetate caused no change in the polarization through the compensating effect of the levulose precipitation. It is needless to add that the double quantity of lead used was beyond that necessary to secure clarification, so that an idea may thus be formed of the probable errors due to excess.

In some interesting clarification experiments by J. A. Hall in the New York Sugar Trade Laboratory the effect of adding varying amounts of dry lead subacetate was studied in another way. Starting with a minimum quantity of the salt, this amount was increased and the effect upon the polarization and the amount of lead dissolved in the clarified filtrate noted. By calculating the dissolved lead to the subacetate it is possible to estimate the dilution, allowing 0.22 cc increase of volume to 1 gram of subacetate as determined by Horne. Only one experiment upon a No. 2 Philippine mat sugar is cited:

A second comparison of effects of varying quantities of clarifying agents on dilution and polarization.

Clarifying agent.	In 100 cc filtrate.		Estimated dilution.	Polarization.
	PbO.	Pb subacetate.		
	Grams.	Gram.	cc.	° V.
Subacetate (cc) ^a	3.0	0.2678		86.70
Dry subacetate (grams).....	0.5	Trace.	Trace.	Too dark to read.
Do.....	1.0	.1530	(0.20)	86.50
Do.....	2.0	.7203	(0.94)	86.60
Do.....	4.0	2.1078	(2.73)	86.50

^a Sp. gr. 1.250.

It will be noted that with an estimated dilution of 0.2 cc instead of a decrease in polarization as would be expected there is an increase. With an estimated dilution of 0.6 cc the reading is the same as that first obtained, so that the combined effect of the dry lead upon the precipitation of levulose and upon the lowering of the rotation of the levulose in solution is seen to be most pronounced. It will be noted that when an excess of dry lead is added not all of this passes into solution. Adding 1 gram excess caused an increase in the filtrate of only 0.74 gram, and 2 grams an increase of only 1.8 grams. After the solution is sufficiently clarified for reading addition of more lead will continue to form a precipitate, so that the rule of adding lead until no more precipitate forms is not always a safe one to follow. An interesting fact in this connection is that the addition of much lead subacetate beyond the point of maximum clarification for low-grade cane products will produce a darkening of the solution. This is due to the well-known color reaction between reducing sugars and alkalis.

If the minimum amount of dry lead subacetate necessary to secure satisfactory clarification be carefully determined for each grade of commercial product and excess beyond this be avoided there is no question but what this method of clarification gives polariscopic readings closer to the true polarization than any other method thus far proposed. The use of dry lead subacetate and subacetate solution as defecating agents in the determination of reducing sugars should of course be avoided.

A third and one of the greatest causes of the lack of agreement in saccharimetric observations between different chemists is variation in temperature. As regards the effect of temperature upon the polarization of pure sucrose nearly all chemists are in very close agreement. For quartz-wedge saccharimeters the researches of Andrews, Wiley, Schönrock, Watts and Tempany, and other chemists show that for each degree Centigrade increase in temperature there is a falling off in the polarization of pure sucrose of about 0.031° Ventzke. The question now arises, with this variation in the specific rotation of sucrose with temperature, what correction, if any, should be applied to the polarization of commercial products.

In my report as associate referee on sugar, made to the association^a in 1905, it was shown that the application of temperature corrections to low grade cane sugars was not advisable, for the reason that the polarization of a sugar is an expression not merely of the sucrose alone but of all the optical constituents present and since some of these optical constituents, more especially the levulose, are affected by temperature in a manner contrary to sucrose, it is not permissible to make a temperature correction for one constituent without at the same time correcting for the others.

This view of the question has been recently contested by Dr. Francis Watts, government chemist, and Mr. H. A. Tempany, assistant government chemist, for the Leeward Islands of the British West Indies, in a recent number of the West Indian Bulletin.^b

^a U. S. Dept. of Agr., Bureau of Chemistry, Bul. 99, p. 20.

^b 1908, 9: 127.

They advocate for the purpose of securing greater uniformity and exactness among analysts the application to all polarizations of a correction formula " $N + 0.00031 tN$ ", where N is the observed reading on the Ventzke scale and t is the difference between the temperature of observation and that at which the polarimeter was standardized." In answer to my criticisms of such a correction when applied to raw cane sugars Messrs. Watts and Tempany reply as follows:

While not disputing the accuracy of the statement concerning the effect of temperature on levulose, we would point out that the process of determining the polariscopic test of a sugar is purely arbitrary and conventional. We take it that the polariscopic test of any sample of sugar is the rotation produced by it when tested in such a way that a sample of chemically pure sucrose tested under precisely similar conditions would give a reading of 100° . The 100 point of the Ventzke, or any other sugar scale, is based on the rotation of a standard weight of sucrose, dissolved in a standard volume of water, at a standard temperature. If at any other temperature this weight of pure sucrose will not give a rotation of 100° on the scale, the scale has been altered; consequently, allowance must be made for this alteration in the scale when polarizing commercial sugars under these conditions.

The above criticism of my previous article is, however, not a valid one. We could say with equal justice: Consequently, allowance must be made for this alteration in the scale when polarizing molasses or honey or condensed milk or glucose or any other substance which is polarized upon a saccharimeter. The only scientific conclusion which could be drawn is—allowance must therefore be made for this alteration in the scale when polarizing pure sucrose; to include commercial sugars and other substances is too sweeping and unwarranted a generalization. It is true that the 100 point of the sugar scale of a saccharimeter is based upon the rotation of a standard weight of c. p. sucrose under certain standard conditions; this sucrose, however, is a means of standardization and nothing more. A definite weight of milk sugar can be made to read 100 upon any saccharimeter and this weight is used for the estimation of milk sugar in milk products. To apply a correction formula for sucrose in such cases would of course be an absurdity.

Quartz may also be used for standardization, and is so used, the 100 point of the French sugar scale being based upon the rotation of a plate of quartz 1 mm thick. It might be said, following the same line of argument as that of Messrs. Watts and Tempany, that because a standard plate of quartz always polarizes 100° irrespective of temperature upon a quartz-wedge saccharimeter, the scale has not been altered and consequently no allowance at all should be taken of temperature in the work of polarization, a conclusion of course perfectly true as regards quartz but not of other substances. Similarly the conclusions worked out for chemically pure sucrose for a given type of saccharimeter are true for chemically pure sucrose but for nothing else, neither for mixtures of sucrose with other substances nor for products which contain no sucrose.

The International Commission for Uniform Method of Sugar Analysis in 1900 decided that it was permissible, as in tropical countries, to adjust saccharimeters to a higher standard temperature than 20°C . This adjustment may be made by changing the quartz wedges of the instrument, by increasing the normal weight of sugar, by increasing the length of the observation tube, or in other ways. When only local comparisons are involved it is advisable and advantageous to make such an adjustment; there is a serious objection, however, against having several separate standards for universal work, since comparisons are no longer possible upon a large class of low-grade saccharine products. Two saccharimeters, for example, one standardized for the rotation of sucrose at 20° and one standardized for the rotation of sucrose at 30° , will give, of course, identical results for pure sucrose, but not for a raw cane sugar, nor for a cane molasses, nor for a large class of other products. Having adjusted our saccharimeter to any desired standard temperature, this standard temperature must be rigidly adhered to if identical observations are to be always obtained between different chemists.

The true polarization then of a raw sugar, as of other saccharine products, is a conventional arbitrary figure representing the sum of the polarizations of the various optical constituents under certain fixed conditions of temperature, weight of substance, volume of solution, length of tube, and quality of light. If the temperature of polarization of a given sugar is different from the standard the correction, if correctly applied, must restore the reading obtained upon this same sugar under standard conditions. Now, the correction advocated by Watts and Tempny and that used by the United States Treasury Department in the Division of Customs will do this for pure sucrose, but it will not do it for a very large class of raw cane sugars for the reasons already given.

Since the publication of my previous paper upon this subject I have had occasion to study the effect of temperature upon the polarization of many sugars and other cane products and have been more thoroughly convinced than ever of the futility of applying such a correction for the purpose of securing greater concordance in the saccharimetric observations of different chemists.

The general results of this work I have condensed into tabular form, showing the ranges of polarization and of reducing sugars for raw cane sugars, and for several types of massecuites and molasses with the corrections necessary to obtain the polarization at standard temperature. The theoretical sucrose corrections according to the formula of Watts and Tempny are appended for purpose of comparison. The values of the table have been made up from averages, some variation was obtained for individual classes of raw sugars, as, for example, those of Louisiana which are very high in reducing sugars and give a correspondingly lower correction. It is believed, however, that the table, on the whole, is a fair average.

Table for correcting polarizations of raw cane sugars, etc., to standard temperature.

[Correction for each °C. above standard temperature.]

Polarization.	Reducing sugars.	Actual correction.	Correction by formula 0.00031 P.
SUGAR.			
° V.	Per cent.	° V.	° V.
100-96	0.00-1.00	+0.028	+0.030
96-94	1.00-1.60	+0.024	+0.029
94-92	1.60-2.20	+0.021	+0.020
92-90	2.20-3.00	+0.017	+0.028
90-88	3.00-3.80	+0.014	+0.028
88-86	3.80-4.60	+0.009	+0.027
86-84	4.60-5.40	+0.005	+0.026
84-82	5.40-6.20	+0.002	+0.026
82-80	6.20-7.00	-0.003	+0.025
80-78	7.00-7.80	-0.007	+0.025
78-76	7.80-8.60	-0.011	+0.024
76-74	8.60-9.00	-0.014	+0.023
MASSECUTE.			
68-72	3.00-10.00	-0.016	+0.022
58-62	12.00-14.00	-0.036	+0.019
44-48	16.00-18.00	-0.057	+0.014
MOLASSES.			
34-30	18.00-20.00	-0.070	+0.010
22-26	24.00-26.00	-0.098	+0.007
16-20	28.00-30.00	-0.116	+0.006

It will be noted that for very high-grade sugars which polarize over 96 an addition of about 0.03° V. for each ° C. increase in temperature will practically restore the reading obtained under standard conditions. The percentage of impurities is too small to affect appreciably the temperature correction for sucrose. As the polarization falls below 96 and the percentage of reducing sugars increases, the effect of the tempera-

ture upon the rotation of the levulose begins to lower the theoretical sucrose correction, until at a point usually about 80 to 86 the two influences—that of the temperature upon the levulose and other impurities and that of the temperature upon the sucrose and quartz wedges of the instrument—counterbalance one another. Two chemists polarizing such a sugar, one working at 30° C. and one working at 20° C., other conditions being equal, will obtain concordant and correct readings; the application of the theoretical sucrose correction would place the observation of the chemist working at 30° C., 0.25° V. too high.

Below 80 the effect of increase in temperature is usually to elevate rather than diminish the reading, this influence becoming more and more pronounced in the massecuites and molasses; the levulose correction more than counterbalances the theoretical one due to sucrose. Every chemist knows how pronounced this influence is on the polarization of sirups and molasses, how the simple handling of the observation tubes will increase the readings. It is the same with low-grade sugars which consist simply of sucrose crystals contaminated with varying amounts of molasses. When such sugars are polarized above 20° C. a correction would have to be subtracted to secure the reading that would be obtained under standard conditions. To add a correction, as required by a sucrose correction formula, would manifestly only further increase the error of observation.

The solution of the temperature question then resolves itself simply into this: If we are to make temperature corrections in the polarizations of commercial products, we must correct for variations in the specific rotation of all the ingredients therein present. If it is impossible to do this, no temperature corrections at all should be applied; instead of this we should strive to make our polarizations as nearly as possible under standard conditions. Custom-house laboratories, arbitration laboratories, and all other laboratories, upon the results of which great interests are involved, should be equipped with cooling and warming apparatus for maintaining a constant uniform standard temperature. The great testing laboratories of Germany are so provided and similar institutions in this country should do as much. For chemists who are unable to provide themselves with this equipment much can be done by moving the laboratory to cooler quarters, as from a hot upper room to a cool basement. By such a change the New York Sugar Trade Laboratory has lowered the temperature of testing from 25° C. to 21.5° C. in hot weather.

The services rendered to science by the researches of the many chemists who have investigated the influence of temperature upon the specific rotation of sucrose are great; the results of their labors are lasting and will stand the test of time. The application, however, of what they have established for pure sucrose to the polarization of all grades of saccharine products is a misapplication. It is a great mistake. It will increase rather than diminish the errors between many of the saccharimetric observations of different analysts and is bound to work great injustice when applied commercially.

A paper on the influence of glycerin, acetanilid, and certain other drugs in the estimation of alcohol by L. E. Warren and H. C. Fuller of the Division of Drugs, Bureau of Chemistry, was presented by Mr. Warren. This work, bearing especially upon the drug investigations, has been printed elsewhere for greater accessibility.^a

The associate referee presented a lengthy paper by S. H. Baer on the colorimetric method for the determination of citral, dealing largely with the chemistry of that substance. The portions on criticisms of the method are reported in abstract.

^a Amer. J. Pharm., 1909, 81: 66.

CITRAL AND ITS ANALYSIS IN TERPENELESS EXTRACT OF LEMON.

By SAMUEL H. BAER.

The analyses were made by three chemists, including the writer, and as all three judged the colors, it would seem that the analyses are as accurate as the colorimetric method permits. Acknowledgment is due S. E. Shaffner for assistance rendered.

Determination of citral in lemon extract by the colorimetric method.

Sample No.	Description.	Citral.	
		Estimated amount present.	Amount found.
		Per cent.	Per cent.
10	Terpeneless oil of lemon solution (dissolved in cologne spirits, 190 proof, or 95 per cent, and colored with lemon peel).....	0.42	0.19
11	Terpeneless oil of lemon solution (dissolved in cologne spirits, 160 proof, or 95 per cent, and colored with turmeric).....	.42	.18
12	Terpeneless oil of lemon solution (dissolved in 38 per cent cologne spirits and city water and filtered through magnesia).....	.42	.10
13	Citral solution (dissolved in cologne spirits of 190 proof, or 95 per cent).....	.42	.40
14	17 pounds oil of lemon, 19 gallons cologne spirits, 23 gallons water (colored with lemon peel and filtered through magnesia).....		.10
15	Alcohol (not cologne spirits, 188 proof, generally used by manufacturers).....		.19
16	Cologne spirits, 190 proof.....		.07
17	50 per cent cologne spirits with city water.....		.07
18	50 per cent cologne spirits filtered through magnesia.....		.08

From these analyses it is seen that when the colorimetric method is applied to the extracts of commerce, the correct result is not obtained. On sample No. 13, a citral solution, the analysis was reasonably close; samples No. 10 and 11 are terpeneless oils of lemon and the low results on citral may be due to the fact that the sample purchased was not pure terpeneless oil of lemon, but a product containing only 50 per cent of the citral that should be there.

Most of the extract manufacturers use 188 proof alcohol, that is, 94 per cent alcohol, which always contains a certain amount of aldehydes, and the sample used in this test, treating the alcohol the same as the lemon extract, showed 0.19 per cent of citral, when there was no citral there at all. If only cologne spirits are used, the results obtained are not so far wrong as if 94 per cent alcohol is used.

Since, therefore, the presence of the impurities in alcohol throw the results off to such an extent; giving too high a per cent of citral, would it not be possible that the impurities in the alcohol at certain times and also in the water, and the very change of one or two ingredients in the lemon oil, might make the result inaccurate, reversing the analysis and showing a smaller per cent of citral than is really present?

The colorimetric method is applicable if the manufacturer used chemically pure citral, distilled water, and aldehyde-free alcohol in the manufacture of his extracts, but such ideal conditions never exist. Further, any manufacturer could discreetly add another aldehyde, even acetaldehyde, to the extent of 0.2 per cent, which would give all the reactions of citral in the extract of lemon by the colorimetric method.

The method is not without use, but if the presence of citral could be determined and estimated quantitatively by a sodium sulphite or carbazone method, then the colorimetric method might be used as a check. Before adopting the colorimetric method as official a committee should be appointed from the association members to test it carefully, under the conditions that the manufacturer must meet since he can not use aldehyde-free alcohol, nor is he always in a position to use distilled water.

Further, suppose the method is accurate, how would the analyses show that the citral used was obtained from lemon oil or the commercial citral obtained from lemon grass oil?

AN OUTLINE TO ASSIST IN THE IDENTIFICATION OF CERTAIN WATER-SOLUBLE COAL-TAR COLORS.

By C. B. COCHRAN.

The reactions given by the coal-tar colors listed in the following outline were all obtained with solutions as dilute as they could be made and still give reactions sufficiently clear and definite to furnish a basis for positive conclusions. Because of the degree of dilution the results here tabulated will, in some cases, appear contradictory to those given by Schultz and Julius. For example, these authors may report a color precipitated by a certain reagent when the precipitation is only partial and therefore does not appear in dilute solutions such as have been used in the preparation of these tables.

The sodium bisulphite reagent is prepared by saturating a 5 per cent solution of sodium hydroxid with sulphur dioxide. The absorption tests with aluminum hydroxid were made by adding between 2 and 3 cc of well-washed aluminum hydroxid (from which the excess of water has been drained through the filter) to 10 cc of the color solution.

The tests with the fuller's earth were made by adding 2 cc of the earth to 10 cc of the color solution. In these absorption tests the aluminum hydroxid and fuller's earth are shaken with the color solution. If, after setting, the supernatant liquid is colorless or very nearly so, the result is recorded as color absorbed. In the majority of cases the results obtained with aluminum hydroxid and fuller's earth are definite and sharp. There are many colors belonging to Class I (Rota's classification) which are much more readily absorbed from their water solutions by aluminum hydroxid than by fuller's earth, while the reverse is true of many colors belonging to Classes II, III, and IV.

In the dyeing tests sodium carbonate was used for making alkaline and hydrochloric acid for acidifying. The alkali solution was very weak and the acid bath about one-half the official strength (1 cc strong hydrochloric acid to 50 cc).

The numbers following the names of the colors refer to the 1904 edition of Green's tables.

COAL-TAR COLORS OF CLASS I.

Solution reduced and in most cases decolorized by stannous chlorid. Original color not restored by hydrogen dioxide.

DIVISION I.—COLOR ABSORBED BY ALUMINUM HYDROXID.

Dye wool red.

SECTION I.—Color precipitated by sodium bisulphite reagent.

Congo red (A) (240) dyes wool and unmordanted cotton red from neutral or faintly alkaline bath, but not from acid bath. Oxalic acid or acetic acid gives a blue precipitate and colorless filtrate.

SECTION II.—Color not precipitated nor solution changed by sodium bisulphite reagent.

Fast red A (102), hydrochloric acid gives a brown precipitate and colorless filtrate. Dyes wool and unmordanted cotton red from acid, alkaline, or neutral bath. Color precipitated by barium chlorid solution.

Azo rubin S (103), color only partially precipitated by hydrochloric acid. Dyes wool red from acid bath but not from alkaline bath. Does not readily dye unmordanted cotton in either bath. Color not precipitated by barium chlorid.

Dyes wool yellow.

Chrysamin R (269), hydrochloric acid gives a brown precipitate, sodium hydroxid a red solution. Barium chlorid and sodium bisulphite reagent each gives a yellow precipitate and colorless filtrate. Dyes wool pale yellow from a neutral bath and unmordanted cotton orange yellow from a neutral or alkaline bath.

Dye wool and unmordanted cotton brown from acid bath.

Bismarck brown (197), decolorized by stannous chlorid and on adding hydrogen dioxid a color somewhat redder than the original color appears. Color precipitated by tannin reagent. Color absorbed from alkaline solution by ether, and on adding dilute acetic acid to the ether solution, the color is taken up by the acid.

Resorcin brown (137), decolorized by stannous chlorid. No color returns on adding hydrogen dioxid. Not precipitated by tannin reagent. Color absorbed by fuller's earth.

DIVISION II.—COLOR NOT ABSORBED OR ONLY PARTIALLY ABSORBED BY ALUMINUM HYDROXID.

Dye wool red in acid bath.

SECTION I.—Sodium hydroxid causes a distinct change in color of water solution.

(1) Sodium hydroxid turns water solution violet.

Ponceau 6 R. B. (169), dyed wool is bluish red, turned blue by hydrochloric acid or sulphuric acid, color in wool dissolves in either acid, giving a blue solution. Hydrochloric acid turns water solution violet, more turns it blue.

(2) Sodium hydroxid turns water solution brown.

Brilliant crocein (146), hydrochloric acid produces little change in color of water solution.

Crystal ponceau (A) (64), dyed wool turned violet by hydrochloric acid and blue by sulphuric acid.

Crocein scarlet 3 B X (104), dyed wool turned red violet by hydrochloric acid or by sulphuric acid.

New coccin (A) (106), color of dyed wool not changed by hydrochloric acid.

(3) Sodium hydroxid turns water solution yellow.

Palatin scarlet (53), dyed wool is scarlet. Color not much changed by hydrochloric acid, but sulphuric acid turns it violet and gives a violet solution. On dilution wool has nearly original color.

SECTION II.—Sodium hydroxid does not cause a distinct change in the color of the water solution.

Group I.—Sulphuric acid turns dyed wool blue or violet and gives a blue or violet solution. Dyed wool is bluish red.

Bordeaux B (A) (65), hydrochloric acid turns dyed wool violet.

Bordeaux S (A) (107), scarlet B. E. E. (P) closely related to Bordeaux S.

Group II.—Sulphuric acid has little or no effect on color of dyed wool. Dyed wool is scarlet.

Ponceau G (A) (55), barium chlorid gives an orange red precipitate, wool dyed orange red.

Ponceau 3 R (A) (56), barium chlorid gives a red precipitate. Dyes wool more red than (55).

Dye wool yellow or orange.

SECTION I.—Hydrochloric acid added to strong acidification precipitates the color or decolorizes the solution (the nitro colors).

Group I.—Color extracted by ether from solution acidified with hydrochloric acid.
Victoria yellow (2).

Martius yellow (3), water solution plus potassium cyanid gives a brown color on warming.

Group II.—Color not extracted by ether from solution acidified with hydrochloric acid.

Naphthol yellow S (4).

SECTION II.—Hydrochloric acid causes a decided change in the color of the water solution (many of the tropæolins).

Group I.—Hydrochloric acid turns dyed wool violet.

Dyed wool is yellow.

Brilliant yellow S (Sch.) (89), dyed wool is yellow turned violet by hydrochloric acid.

Metanil yellow (Sch.) (95), dyed wool is orange yellow turned violet by hydrochloric acid.

Group II.—Hydrochloric acid turns dyed wool brown.

Chrysoidin R (18). This color is absorbed by fuller's earth and partially absorbed by aluminum hydroxid.

Group III.—Hydrochloric acid turns dyed wool red.

Fast yellow (8).

SECTION III.—Color of water solution not decidedly changed by hydrochloric acid. (If a precipitate appears only a part of the color is precipitated.)

Dyed wool is yellow.

Naphthol yellow S (4), dyed wool is decolorized by hydrochloric acid.

Tartrazin (94), color of dyed wool not changed by hydrochloric acid.

Dyed wool is yellow orange to orange.

Tropæolin O (84).

Tropæolin OOO (85).

Orange G (14).

Dyed wool is red orange.

Mandarin G (86), dyed wool is turned red violet by hydrochloric acid or sulphuric acid.

Ponceau 4 G. B., color of dyed wool not changed by hydrochloric acid nor by sulphuric acid.

COLORS OF CLASS II.

Solution decolorized by stannous chlorid, original color returns on addition of hydrogen dioxid. (Bismarck brown, which might be referred to this class, is included under Class I.)

(1) Dyes wool and cotton bluish red (most readily from an alkaline bath).

Safranin (584), much hydrochloric acid turns water solution blue violet. Color absorbed by fuller's earth, precipitated by tannin reagent. Sulphuric acid turns dyed wool green, solution green, hydrochloric acid blue.

(2) Dyes wool blue from alkaline or neutral bath, cotton a paler blue from neutral bath.

Methylene blue (650), color absorbed by fuller's earth precipitated by tannin; hydrochloric acid turns dyed wool robin's-egg blue, sulphuric acid green.

COLORS OF CLASS III.

Stannous chlorid produces no further effect on the color than hydrochloric acid. Sodium hydroxid produces a precipitate or decolorizes the solution. All the colors given in this class except auramin (425) are decolorized by sodium bisulphite reagent.

The color reappears on heating and disappears on cooling. With the exception of acid magenta (A) (462) they are all absorbed by fuller's earth.

Dye wool red.

(1) Dye wool from acid bath only, do not dye unmordanted cotton in either bath.

Acid magenta (462), color absorbed by aluminum hydroxid. Dyed wool is decolorized by hydrochloric acid, sulphuric acid, sodium hydroxid, or ammonium hydroxid. Tannin reagent gives no precipitate.

(2) Dyes wool and also unmordanted cotton most readily from a neutral bath.

Fuchsin (448), color not absorbed by aluminum hydroxid. Dyed wool turned red brown by hydrochloric acid or sulphuric acid. Tannin reagent gives a precipitate.

(3) Dyes wool yellow from neutral or alkaline bath. Does not dye unmordanted cotton. Aurantin (425).

Dye wool green.

Dye from acid bath: Guinea green B (A) (433) and acid green (434) do not dye cotton.

Dye from neutral or alkaline bath: Ethyl green (428) dyes unmordanted cotton more readily than malachite green.

Malachite green (427), dyed wool is blue green, turned at first grass green by hydrochloric acid or sulphuric acid, then yellow; on dilution, blue.

Dye wool violet from neutral or alkaline bath.

Methyl violet (451), sodium hydroxid gives a brown precipitate and brown solution.

Ethyl violet (453), sodium hydroxid gives a white precipitate, colorless on warming. Either dyes unmordanted cotton from alkaline bath.

Dyes wool blue from acid bath.

China blue (480), color absorbed by aluminum hydroxid. Solution decolorized by sodium bisulphite reagent. Color does not readily return on heating, but does return on adding a drop of hydrochloric acid. Dyed wool decolorized by ammonium hydroxid, turned reddish brown by sulphuric acid.

COLORS OF CLASS IV.

Colors not reduced by stannous chlorid. Solution not decolorized and color not completely precipitated by sodium hydroxid.

Dye wool red from neutral bath.

Dyed wool is red orange to orange red:

Eosin (512), color not absorbed by fuller's earth nor by aluminum hydroxid. Water solution yellow to orange with green fluorescence. Hydrochloric acid or sodium bisulphite reagent gives an orange precipitate.

Dye wool bluish red from neutral bath:

(a) Color completely absorbed by fuller's earth. Sodium bisulphite reagent gives no precipitate, but causes only a loss of fluorescence.

Rhodamin G (502), water solution red violet with red fluorescence.

Rhodamin B (504), water solution bluish red with orange brown fluorescence.

(b) Color only partially absorbed by fuller's earth. Sodium bisulphite reagent precipitates the color.

Erythrosin (516), water solution cherry red. (Green's tables give no fluorescence. A sample marked "Grübler" gave green fluorescence.) Hydrochloric acid gives an orange brown precipitate. Sodium bisulphite reagent gives an orange-red precipitate.

Rose bengal (520), water solution cherry red. No fluorescence. Hydrochloric acid gives a brown-red precipitate. Sodium bisulphite reagent a pink precipitate.

Phloxin (521), water solution bluish red with green fluorescence. Hydrochloric acid gives an orange precipitate. Sodium bisulphite a pink precipitate.

**OFFICERS, REFEREES, AND COMMITTEES OF THE ASSOCIATION
OF OFFICIAL AGRICULTURAL CHEMISTS FOR THE YEAR 1908-9.**

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Vice-president.

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Secretary.

H. W. WILEY, Washington, D. C.

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Nitrogen:

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Separation of nitrogenous bodies: P. F. Trowbridge, Columbia, Mo. (meat proteids).

Potash: B. B. Ross, Auburn, Ala.

Soils: S. D. Averitt, Lexington, Ky.

Dairy products: J. M. Bartlett, Orono, Me.

Foods and feeding stuffs: J. P. Street, New Haven, Conn.

Food adulteration: H. E. Barnard, Indianapolis, Ind.

Sugar: A. H. Bryan, Washington, D. C.

Tannin: F. P. Veitch, Washington, D. C.

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Jas. A. Bizzell, Ithaca, N. Y. (special associate referee on available potash).

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Fruit products: C. B. Cochran, Westchester, Pa.

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Flavoring extracts: E. M. Chace, Washington, D. C.

Spices: A. F. Seeker, New York, N. Y.

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Fats and oils: T. J. Bryan, Chicago, Ill.

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Cereal products: E. F. Ladd, Agricultural College, N. Dak.

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Tea and coffee: A. G. Woodman, Boston, Mass.

Preservatives: P. B. Dunbar, Washington, D. C.

Determination of water in foods: P. F. Trowbridge, Columbia, Mo.

Sugar:

Molasses methods: H. P. Agee, New Orleans, La.

Chemical methods: A. H. Bryan, Washington, D. C.

Tannin: C. F. Sammet, Washington, D. C.

Insecticides: Howard R. Watkins, Washington, D. C.

Inorganic plant constituents: O. M. Shedd, Lexington, Ky.

Medicinal plants: {C. H. La Wall, Philadelphia, Pa.
H. H. Rusby, New York, N. Y.

Water: W. W. Skinner, Washington, D. C.

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Committee on the unification of methods of analysis of fats and oils.

Mr. L. M. Tolman, Washington, D. C., chairman.
 Mr. P. H. Walker, Washington, D. C.
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Standing committee on recommendations of referees and revision of methods.

(The figures in parentheses refer to number of years appointee is to serve.)

Committee A: B. B. Ross (3), J. P. Street (2), J. K. Haywood (1), chairman, *Bureau of Chemistry, Washington, D. C.*

Committee B: E. M. Chace (3), F. W. Woll (2), chairman, *Agricultural Experiment Station, Madison, Wis.*; F. P. Veitch (1).

Committee C: C. D. Howard (3), A. L. Winton (2), chairman, *U. S. Food Inspection Laboratory, Chicago, Ill.*; L. M. Tolman (1).

CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This association shall be known as the Association of Official Agricultural Chemists of North America. The objects of the association shall be (1) to secure uniformity and accuracy in the methods, results, and modes of statement of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State, Provincial, or National agricultural experiment station or agricultural college, or with any State, Provincial, or National institution or body in North America charged with official control of the materials named in section 1, shall alone be eligible to membership; and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to enter motions or vote in the association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. All persons eligible to membership shall become members ex officio and shall be allowed the privileges of membership at any meeting of the association after presenting proper credentials. All members of the association who lose their right to such membership by retiring from positions indicated as requisite for membership shall be entitled to become honorary members and to have all privileges of membership save the right to hold office and vote. All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote.

(3) The officers of the association shall consist of a president, a vice-president, and a secretary, who shall also act as treasurer; and these officers, together with two other members to be elected by the association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board whose members are eligible to membership, his office shall be considered vacant, and a successor may be appointed by the executive committee, to continue in office till the annual meeting next following.

(4) There shall be appointed by the executive committee, at the regular annual meeting, from among the members of the association, a referee and such associate referees for each of the subjects to be considered by the association as that committee may deem appropriate.

It shall be the duty of these referees to prepare and distribute samples and standard reagents to members of the association and others desiring the same, to furnish blanks for tabulating analyses, and to present at the annual meeting the results of work done, discussion thereof, and recommendations of methods to be followed.

(5) The special duties of the officers of the association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this association shall be held at such place as shall be decided by the association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

(7) No changes shall be made in the methods of analysis used in official inspection, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of the particular inspection affected to test the proposed changes.

(8) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(9) The executive committee will confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the association.

(10) All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by the approval of two-thirds of the members present entitled to vote.

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BUREAU OF CHEMISTRY--BULLETIN No. 123.

H. W. WILEY, Chief of Bureau.

METABOLISM OF ORGANIC AND INORGANIC PHOSPHORUS:

A FEEDING EXPERIMENT USING PHYTIN
AND SODIUM PHOSPHATES.

BY

F. C. COOK,
PHYSIOLOGICAL CHEMIST.



WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1909.

LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., January 15, 1909.

SIR: I have the honor to submit for your inspection and approval a report on a phosphorus metabolism experiment conducted by F. C. Cook under the supervision of the Chief of Bureau. The report covers an experiment in rabbit feeding, extending over a period of six months, during which organic and inorganic phosphorus were fed, and includes calcium, magnesium, and total and ether-alcohol soluble phosphorus balances. At the conclusion of the experiment, complete analyses were made of the bodies of the rabbits, also of normal rabbits, which furnish some valuable data. Although the number of experiments is limited, the complete review of the literature bearing on the subject, which is included in this paper, greatly enhances its value and the interest both in this country and abroad in the relative value of the organic and inorganic forms of phosphorus, iron, etc., in the body economy makes the issuance of this contribution on the subject advisable.

I recommend that the manuscript be published as Bulletin 123 of the Bureau of Chemistry.

Respectfully,

II. W. WILEY, *Chief.*

HON. JAMES WILSON,
Secretary of Agriculture.

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METABOLISM OF ORGANIC AND INORGANIC PHOSPHORUS.

REVIEW OF THE LITERATURE.

Much work has already been done on phosphorus metabolism, both in regard to the inorganic and organic forms of phosphorus, and many investigations have been recorded showing the advantages of the various organic forms, such as lecithin, glycerophosphoric acid, phytin, etc. Most of this work has been done abroad, although some has been published in this country, notably the researches of Jordan, Patten, and Hart;^a Mendel and Underhill;^b and Le Clerc and Cook.^c It seemed advisable, therefore, to present a general survey of the contributions previously made on this mooted question.

PHOSPHORUS COMPOUNDS.

In speaking of phosphorus compounds, Bunge^d states that certain of them probably should be regarded as essential organic food substances for man; also that in all animal and vegetable tissues, in every cell are found two complex organic compounds which are rich in phosphorus, namely, the lecithins and the nucleins.

According to the recent recommendations of the joint committee of the American Physiological Society and the American Society of Biological Chemists on protein nomenclature, the word "proteid" should be abandoned and the word "protein" should designate that group of substances which consists essentially of combinations of α -amino acids and their derivatives.

The conjugated proteins are divided into (a) nucleoproteins, (b) glycoproteins, (c) phosphoproteins, (d) hemoglobins, (e) lecithoproteins. The nucleoproteins are compounds of one or more protein molecules with a nucleic acid. The phosphoproteins are compounds of the protein molecule with some, as yet unidentified, phosphorus-containing substance other than a nucleic acid or lecithin. The lecithoproteins are compounds of the protein molecule with lecithins (lecithans, phosphatids).

^a Amer. J. Physiol., 1906, 16 : 268.

^b Ibid., 17 : 75.

^c J. Biol. Chem., 1906, 2 : 203.

^d Physiologic and Pathologic Chemistry, 2d ed., 1902.

LECITHINS.

The lecithins are ester compounds which may be regarded as having been formed by the union of one molecule of glycerol with two molecules of a fatty acid (stearic acid, palmitic acid, or oleic acid), one molecule of phosphoric acid, and one molecule of cholin, with the loss of four molecules of water. The formula for lecithin is $C_{44}H_{90}NPO_6$. The lecithin radical contains one atom of nitrogen for every atom of phosphorus.

Cholin is an ammonium base, the composition of which is accurately known. When heated it splits into glycol (ethylene alcohol), and trimethylamin. Its synthesis corresponds with this decomposition. Wurtz ^a produced it by the action of ethylene oxid and water on trimethylamin. In the animal kingdom cholin has, up to the present time, been found only in lecithin. It was first obtained by Strecker ^b from the bile, which contains lecithin, and hence was called cholin. Liebreich ^c found it among the products of the decomposition of phosphorus compounds from brain tissue. Diaconow ^d showed that it was a product of the decomposition of lecithin. In the new tissues of plants cholin is found in other combinations as well as in lecithin. At present but little is known about the part which the lecithins play in the vital functions.

An important question is whether the lecithins of the body tissues are produced from the lecithins of the food or by synthesis from other materials such as fat, protein, and phosphoric acid. It has been ascertained from experiments on artificial pancreatic digestion that the lecithins take up water and readily split up into glycerophosphoric acid, fatty acids, and cholin. It is not yet known whether this decomposition is complete in normal digestion, or a portion is absorbed unchanged, and if so, how large a portion; whether only the undecomposed part, when absorbed, can be utilized in the building up of the tissues, or the products of decomposition which are absorbed again become united; or finally whether lecithin may also be formed from other material. The absorption of lecithin or of its products of decomposition is complete, according to Bunge, as he states that neither lecithin nor glycerophosphoric acid can be found in the feces. More recent work, however, by Long ^e seems to show that the feces sometimes contain lecithin in considerable quantities. The presence of lecithin in milk, eggs, and many other foods indicates that this substance is essential in nutrition.

^a Centrbl. med. Wissensch., 1868, 6 : 69, 431.

^b Ann. Chem. Pharm., 1862, 123 : 353; 1868, 148 : 77.

^c Ibid., 1865, 134 : 29.

^d Centrbl. med. Wissensch., 1868, 6 : 97, 434.

^e J. Amer. Chem. Soc., 1906, 28 : 704; Long and Johnson, *ibid.*, 1499.

NUCLEO-PROTEINS.

By this name are designated those compound proteins which yield true nucleins on pepsin digestion and which, on cleavage with alkali, yield protein and nucleic acid. The nucleo-proteins seem to be widely distributed in the animal body. They occur chiefly in the cell nuclei, but they also often occur in the protoplasm. They may pass into the animal fluids on the destruction of the cells; hence nucleo-proteins have also been found in blood serum. They may be considered as combinations of a protein nucleus with a side chain which Kossel^a calls the "prosthetic group." This side chain, which contains the phosphorus, yields on the decomposition of many nucleo-proteins, such as that from the yeast cell^b or from the pancreas,^c besides nuclein bases, also reducing substances, which form crystalline combinations with phenyl-hydrazin. The nucleo-proteins contain from 0.5 to 1.6 per cent of phosphorus.

The nucleo-proteins split into a nuclein and an albumin radicle and the nuclein radicle is further split into nucleic acid and albumin. The nucleic acids on cleavage yield in addition to the purin bases three simple pyrimidin derivatives, uracil, cytosin, and thymin. In a recent article by Osborne and Heyl^d it appears that all but one-sixteenth of the nitrogen of nucleic acid probably belongs to guanin, adenin, cytosin, and uracil, of which one molecule of each is present for every four atoms of phosphorus.

It is important to distinguish between the nucleo-proteins and the pseudo nucleo-proteins. The latter bodies are obtained as an insoluble residue on digestion of certain nucleo-albumins or phospho-glyco-proteins with pepsin hydrochloric acid. They contain phosphorus but yield no nuclein bases. Among the pseudo nucleo-proteins may be mentioned phospho-proteins and lecitho-proteins. These substances are often fed in the form of casein or vitellin in metabolism experiments.

NUCLEINS.

The generic name of nuclein has been bestowed upon a large number of very different organic phosphorus compounds, which are to be found in all animal and vegetable tissues, being especially abundant in the nuclei of cells. The nucleins contain about 5 per cent of phosphorus and are formed by the cleavage of nucleo-protein. The nucleins are acids, and the phosphorus is given off as phosphoric acid on boiling with water, and more rapidly on boiling with alkalies or acids. But the organic substances which are combined with the

^a Arch. Anat. Physiol., Physiol. Abt., 1893, p. 157.

^b Ibid., 1891, p. 359.

^c Hammarsten, Zts. Physiol. Chem., 1894, 19 : 19.

^d Amer. J. Physiol., 1908, 21 : 157.

phosphoric acid appear to be of varying characters. Most nucleins are protein compounds, although a few do not contain protein. Nucleins appear to occur mostly in the tissues, not in a free state, but as compounds with protein as nucleo-albumins, and perhaps also with lecithin, and the gastric digestion separates them from these bodies.

Whether the nucleins of the body tissues arise from the nucleins of food (in which case they would rank among the number of essential food substances), or whether the nucleins are formed in the body by synthesis, is a question of great importance, about which, as in the case of the mode in which the lecithins originate, very little is known. The extensive observations by Miescher ^a on Rhine salmon seem to show that the nucleins as well as the lecithins arise in the animal body by synthesis.

PHOSPHO-GLUCO-PROTEINS.

This group includes the phosphorized gluco-proteins. These compound proteins are decomposed by pepsin digestion and split off para- or pseudo-nuclein substances, similar to nucleo-albumins. They differ from the nucleo-albumins in that they yield a reducing substance on boiling with acids, and from the nucleo-proteins in that they do not yield purin bases.

Only two phosphorized gluco-proteins are known at the present time. Ichthulin, which occurs in carp eggs and was studied by Walter, ^b was considered by him as vitellin for a time. In regard to solubilities, ichthulin behaves like a globulin. Walter prepared a reducing substance from the para-nuclein of ichthulin, which gave a crystalline combination with phenylhydrazin. The other phospho-gluco-protein is helico-protein, obtained from the glands of the small snail *Helix pomatia*.

INORGANIC PHOSPHORUS.

In regard to phosphoric acid Hammarsten ^c states that there seems to be no doubt that its importance lies chiefly in the fact that it takes part in the formation of nucleins and thereby indirectly makes possible the processes of growth and division which are dependent upon the cell nuclei. Loew ^d has shown, by means of cultivation experiments on the alga *Spirogyra*, that only by supplying phosphates (in this case potassium phosphate was used) was the nutrition of the cell nucleus made possible, and thereby the growth and division of the cells. The cells of the *Spirogyra* can be kept alive, and indeed produce

^a Cited in Hammarsten's Textbook of Physiological Chemistry, New York, 1908.

^b Zts. physiol. Chem., 1891, 15 : 477.

^c Physiological Chemistry, 2d ed., 1898.

^d Biol. Centrbl., 1891, 11 : 269.

starch and proteins for some time, without a supply of phosphates, but their growth and propagation suffer. Phosphoric acid is also without doubt of importance in the formation of the lecithins and other organic phosphorus compounds. The inorganic forms of phosphorus occur in the bones and teeth as calcium phosphate and magnesium phosphate.

A small part of the phosphorus of the food is in the form of inorganic salts, as in meat, but is mostly in organic combination, as in milk, eggs, etc., as nucleo-albumin, nucleins, casein, lecithin, and vitellin.

PHOSPHORUS METABOLISM.

Röhmnn ^a and his followers, Marcuse, ^b Steinitz, ^c Leipziger, ^d Zadik, ^e Ehrlich, ^f and Gottstein, ^g favor the organic forms of phosphorus, and the opinion of the majority is that the nucleins, not being easily split by the digestive juices, are absorbed with difficulty; consequently the body builds its organic compounds from the more simple organic phosphorus bodies.

The theory of Salkowski, Umber, and the Breslau school is that the body has not the power to build from phosphorus-free protein and inorganic phosphates the organic phosphorus combinations essential to the life of the cell. Lack of phosphates in the food is without influence on phosphorus retention, and excessive feeding of organic phosphorus causes the retention of more phosphorus than the excessive feeding of inorganic phosphates. The advantages of organic phosphorus over inorganic phosphates during the period of growth is shown by Cronheim and Müller ^h by experiments performed on five infants and a boy. The problem was to determine whether the two forms of organic phosphorus, protein-phosphorus and fat-phosphorus, exert the same influence on the assimilation of phosphorus and nitrogen. The foods used were a casein preparation from skimmed milk and a lecithin preparation from the yolk of eggs. The food containing lecithin appeared to favor the retention of calcium, most of which was found in the bones. Experiments were also made upon five dogs, four weeks old at the beginning of the experiment. The food given consisted of milk, rice, flour, and butter. Three dogs received this diet plus egg yolk, and two received plasmon

^a Berlin. klin. Wochenschr., 1898, 35 : 789.

^b Arch. gesam. Physiol., 1896, 64 : 223.

^c Ibid., 1898, 72 : 75.

^d Ibid., 1899, 78 : 402.

^e Ibid., 1899, 77 : 1.

^f Stoffwechselversuche. Inaug. Diss., Breslau, 1900.

^g Ibid., 1901.

^h Zts. diät. physik. Therapie, 1903, 6 : 25.

together with sodium phosphate. The amount of food given was calculated according to the following formula: $(\sqrt[3]{\text{Body weight}})^2$. One dog which was fed on egg yolk died, the histological section showing that death was due to pneumonia. There was no difference in the appearance of the dogs and all grew equally well. The marrow of the bones of the dogs fed on egg yolk was yellow and richer in fat, while the marrow of the bones of the plasmon-fed dogs was red and richer in blood but poorer in fat. On aging, the red marrow became yellow, proving that the dogs which were fed with egg yolk made more progress.

The same experiment was tried with four guinea pigs and one of those fed on egg yolk died of pneumonia in three months. The pigs so fed also showed fatty livers, which weighed more than the other livers. The increase in weight was greater in the case of the pigs fed on egg yolk than in the case of those which were fed plasmon. In all cases the phosphorus content of the brain was the same.

The general conclusion was that the growth of nitrogenous tissue is facilitated if phosphorus is ingested in the form of egg yolk; that is, in organic form. The daily amount of phosphorus needed by the average man, according to Siven,^a is from 0.7 to 0.8 gram, and according to Ehrström,^b from 1 to 2 grams. He states that phosphorus is necessary for the proper nourishment of the bones, nervous system, body proteins and cells, and that the body strives to retain the phosphates more than other salts. Other investigations along this line were carried out by Tigerstedt,^c Renvall,^d and Schlossmann.^e

Slowtzoff,^f in studying the action of lecithin on metabolism, found a plus nitrogen balance accompanied by a diminished excretion of phosphorus and also of purin bases. Where the nitrogen balance was minus, the case could be otherwise explained.

Loewi^g investigated the metabolism of nucleins. He experimented on himself and found that a part of the nuclein was split in the intestine, the phosphorus of the split portion going into the feces, while the nitrogen was absorbed. The part not split was nearly all absorbed and consequently the phosphorus remained in organic combination. It is possible by nuclein feeding to bring the body into the same nitrogen and phosphoric-acid relation as exists in the nucleins themselves, since nuclein ingestion increases the retention of nitrogen and slightly increases that of the phosphorus.

^a Skand. Arch. Physiol., 1901, 11 : 308.

^b Ibid., 1903, 14 : 82.

^c Ibid., 1904, 16 : 67.

^d Ibid., 1904, 16 : 91.

^e Arch. Kinderheilk., 1905, 40 : 1.

^f Beitr. chem. Physiol. Path., 1906, 8 : 370.

^g Arch. exper. Path. Pharm., 1900, 44 : 1; 1901, 45 : 157.

Jacob and Bergell^a studied the influence of nuclein food on the blood and metabolism and found that it increases the number of the leucocytes. Brücke^b conducted experiments to show that the benefit derived from egg yolk was due to lecithin. Danilewsky^c determined that lecithin had great influence on the growth of young animals. Umikoff^d at about the same time showed that rats and doves died when fed on a phosphorus-free diet and also when fed on an inorganic phosphorus diet plus egg albumin, and barely lived on a nuclein-phosphorus diet, but thrived when lecithin was fed. Selensky^e also demonstrated the valuable effects of lecithin. Serono^f was the first to inject lecithin into a human subject, and the experiment gave favorable results. Danilewsky showed that lecithin increased the number of red blood corpuscles and the hemoglobin; also that the appetite, body weight, and growth increased. Moreover, the resistance of the body to disease was greater after lecithin feeding, and the loss of body weight during hunger was decreased. Wildiers,^g however, did not get results corroborating Danilewsky.

Desgrez and Zaky^h experimented with guinea pigs and dogs and good results were obtained for four and one-half months after the lecithin feeding was stopped. In the urine there was more nitrogen but less phosphorus than in the controls. A larger part of the urine nitrogen was excreted as urea in lecithin-fed animals and a more complete destruction of the protein was brought about in these cases.

Gilbert and Fournier,^h Carrière,ⁱ Claude and Zaky,^j and others carried on clinical experiments with lecithin and found a resultant increase in appetite, number of red corpuscles, hemoblasts, and hemoglobin.

Gliken^k made a study of the lecithin content of young animals born blind and helpless and of birds, eggs, etc. He states that the very young animals show a higher lecithin content than do mature animals; that the lecithin content decreases with the growth and age of the animal, and that the young animals come into the world with a large relative amount of lecithin in their bodies.

^a Zts. klin. Med., 1898, 35 : 171.

^b Vorlesungen über Physiologie, 2d edition, 1875, 1 : 270.

^c Compt. rend., 1895, 121 : 1167.

^d Biology of Phosphorus, Diss., St. Petersburg, 1895.

^e Cited by Gilbert and Fournier, Compt. rend. soc. biol, 1901, 53 : 145.

^f La cellule, 1900, 17 (2) : 385.

^g Compt. rend., 1904, 139 : 819.

^h Compt. rend. soc. biol., 1901, 53 : 145.

ⁱ Compt. rend., 1901, 133 : 314.

^j Gaz. hospitaux civils militaires, 1901, No. 113, p. 1084.

^k Biochem. Zts., 1907-8, 7 : 286.

Nerking^a studied the lecithin distribution in animal organisms, and quotes the lecithin content of the organs of various animals as varying from 0.55 per cent in the pancreas to 1.5 per cent in the liver. Schulze^b investigated the lecithin content of various plant seeds, and found from 0.5 to 1.5 per cent. This author also determined the lecithin content of various portions of the bodies of rabbits, from which it appeared that the average lecithin content equaled 0.45 per cent of the living weight of the rabbits. In the case of a hedgehog the average per cent of lecithin was 0.82 per cent of the live weight. A study of the stability of egg and brain lecithins has recently been made by Long^c and a further study of lecithin emulsions was made by Long and Gephart.^d

In making determinations of the deposition of lecithin and its content in organisms Franchini^e found that feeding lecithin to rabbits increased the content of this substance and also of glycerophosphoric acid in the liver and the muscles, but not in the brain. Lecithin remains in the liver sometimes for fifteen days after its ingestion has been stopped. The feeding causes a slight increase of glycerophosphoric acid and of formic acid but not of cholin. Most of the ingested lecithin is absorbed, since only a very small increase is noted in the feces.

According to observations made by Merservizky,^f lecithin forms 15.35 per cent of fresh hens' eggs. After six days the lecithin content diminishes. The lecithin of the yolk is a storehouse of food for the developing germ, and is used in the development of the skeletal phosphoric acid, in the building up of the phosphorus of proteins, and for the liberation of energy, after which the fat radical is oxidized.

According to Küttner,^g the influence of lecithin on the activity of the digestive ferments varies with different enzymes, having a favorable effect upon the activity of the gastric and pancreatic enzymes, but a retarding effect upon others. How lecithin itself is affected he could not determine.

Koch and Reed,^h in an article on the relation of the extractive to the protein phosphorus in the *Aspergillus niger*, express the view that protein, or in the case of *Aspergillus niger*, nuclein phosphorus is the most important form of phosphorus for cell life. It is formed at the expense of the other forms of phosphorus, excepting lecithin, and its formation is not diminished even in extreme starvation. In building up the nucleins lecithin probably takes no direct part. When lecithin is metabolized some or all of its phosphoric acid may be built up into nucleins as a matter of economy to the organism. The

^a Biochem. Zts., 1908, 10 : 193.

^b Zts. physiol. Chem., 1908, 55 : 338.

^c J. Amer. Chem. Soc., 1908, 30 : 881.

^d Ibid., p. 895

^e Biochem. Zts., 1907, 6 : 210.

^f Russky Uratch, 1907, No. 9, p. 302.

^g Zts. physiol. Chem., 1906-7, 50 : 472.

^h J. Biol. Chem., 1907, 3 : 49.

extractive, water-soluble forms of phosphoric acid are the ones from which the others are built and represent the intermediary steps between the phosphates and the more complex phosphorus combinations.

Kalaroukoff and Terroine^a studied the influence of lecithin on the action of the pancreatic lipase and found very little, if any, increased activity when lecithin was present.

Scott,^b in his experiment on phosphorus liberation from nuclein compounds, determined that it is more difficult to cause phosphorus to pass from its nucleic acid combination to an inorganic condition than has been supposed.

Michel^c investigated the quality of woman's milk and found that the utilization of its nutritive materials by infants is nearly complete. The salts were least utilized, 40 per cent of calcium and 10 per cent of phosphoric acid being rejected in the feces.

Keller^d studied the metabolism of phosphorus by determining the phosphoric acid in the urine of infants fed with woman's and with cow's milk, and found less phosphoric acid so excreted in the case of the breast-fed children. Whether this was due to a greater excretion in the feces or to a better assimilation of the phosphorus of the mother's milk remains to be determined.

In the experiments carried out by Jordan, Hart, and Patten,^e it was found in the case of cows fed on a high phytin diet that when the amount of phytin fed was reduced the amount of fat in the milk was reduced, although there was no effect on the total solids and casein. There was also a smaller excretion of urine and a tendency to constipation. In these experiments there was a considerable loss of body phosphorus for days, when the cows were fed on a low phosphorus diet, with no apparent ill effects. The amount of phosphorus in the egesta was affected but little by the changes of the phosphorus fed; the organic phosphorus was the portion affected, if any effects were produced at all.

Some experiments by McCollum and Hart^f indicate that the liver and blood have the property of cleaving the salts of phytic acid with the production of inorganic phosphoric acid. The wide distribution of inositol in the tissues makes it impossible to say as yet whether it is also a product of this cleavage. These results are in accord with those of Mendel and Underhill,^g who showed that the intestine is not necessarily involved in the excretion of the metabolic products of phytin in certain animals, and also with the conclusions of Scofone,^h that the enzymes of the digestive tract do not alter phytin. Exami-

^a Compt. rend. soc. biol., 1907, 63 : 372.

^b Brit. Med. J., 1906, (2) p. 1791.

^c L'obstetrique, Paris, 1897, 2 : 518.

^d Abs., Chem. Centrbl., 1899, 70 : 54.

^e Amer. J. Physiol., 1906, 16 : 268.

^f J. Biol. Chem., 1908, 4 : 497.

^g Amer. J. Physiol., 1906, 17 : 75.

^h Abs., Biochem. Centrbl., 1905, 3 : 606.

nations of the action of ptyalin, pepsin, and trypsin have confirmed Scofone's results.

Experiments made with extracts of muscle and kidney did not give results which pointed toward the presence of a phytase in these tissues.

Suzuki and Yoshimura^a studied the distribution of anhydroxymethylene phosphorus (phytin), and give a method for extracting the compound, which is a calcium or a magnesium salt. In the juice of tubers and fruit more inorganic than organic phosphorus is found.

Suzuki, Yoshimura, and Takaishi^b made an investigation of the enzyme which decomposes anhydroxymethylene diphosphoric acid, and state that when rice bran and water are allowed to stand the organic compound will be decomposed and the amount of soluble inorganic phosphoric acid increased. When boiled this action does not take place. The same change takes place when barley and rape seeds are used. No other enzyme will do this.

As opposed to the beneficial results of organic phosphorus Keller^c got very favorable results from feeding normal milk plus inorganic phosphates.

Kochmann^d studied the changes in the inorganic constituents in the tissues of rabbits poisoned by phosphorus. He made iron, calcium, magnesium, phosphorus, potassium, and sodium estimations in the liver, heart, muscles, and bones and compared them with similar estimations in normal animals. His conclusions are that a definite effect was produced on phosphorus metabolism and that the use of phosphorus in bone affections and as a stimulant is well founded. Calcium, potassium, and sodium replace one another. The magnesium metabolism is also affected in the cases of phosphorus poisoning, and the excretion of phosphorus and calcium run parallel.

More recent work by Hart and McCollum^e on feeding inorganic phosphates to growing pigs has been conducted for two years. According to the abstract published by the authors, the results clearly indicate that inorganic phosphates, such as bone ash, finely ground rock phosphate, or precipitated calcium phosphate (a mixture of di- and tri-calcium phosphates) can be used by these animals in connection with rations containing insufficient phosphorus. Young animals of 40 pounds weight, receiving inorganic phosphates, together with other salts as supplementary to a ration very low in mineral constituents, grew to be animals of 280 pounds weight,

^a Abs., Chem. Centrbl., 1907, 78: 1636.

^b Ibid., 1637.

^c Abs., Zts. diät. physik. Therapie, 1901, 5: 147.

^d Arch. gesam. Physiol., 1907, 119: 417.

^e Abs., Science, 1908, 28: 217.

and bore litters of fairly vigorous pigs, which on the same ration completed the cycle back to 80 pounds, while animals on the same ration, without the inorganic phosphates, collapsed in three months, losing weight and the use of their legs. Other important observations made are as follows: (1) Animals on a ration extremely low in phosphorus made as large gains, up to 75 to 100 pounds, as did animals receiving an abundance of this element, but after reaching this point the weight was reduced and collapse followed. (2) When such low phosphorus rations as induced these symptoms were supplemented by inorganic phosphates, no unfavorable results appeared. Animals fed a low phosphorus diet, supplemented by inorganic phosphates, made as vigorous a development as other animals receiving all the phosphorus in the organic form. (3) Determination of calcium and phosphorus in the principal organs and tissues of the animals fed on the low phosphorus ration showed that they maintained their normal body composition. The per cent of ash in the skeleton of pigs on a depleted phosphorus ration was reduced to nearly one-half that of pigs which received a normal ration, or the phosphorus-poor ration plus inorganic phosphates. When the animals were starving for phosphorus they derived it from their bones, but always removed calcium and phosphorus in the proportions found in tricalcium phosphate.

PHOSPHORUS ELIMINATION.

In studying the absorption and elimination of phosphorus and other substances the influence of the reactions of the gastro-intestinal tract is of great importance, as is the character of the ash of the food ingested. All of the conditions influencing acidity and alkalinity affect the absorption and the path of excretion of phosphorus, calcium, and magnesium salts. In the case of herbivora a large portion of these elements is eliminated in the feces, no absorption having taken place. This is likely to happen when the food ash is alkaline and sufficient calcium and magnesium are present to combine with the phosphoric acid. There is then an excretion of these elements through the intestines as well as through the kidneys, and when alkalis are in excess of acids in the gastro-intestinal tract the elimination through the bowel is likely to exceed that through the urine. In omnivorous animals a larger portion of the phosphorus, calcium, and magnesium, as well as the nitrogen, is eliminated by the kidneys than is the case with herbivorous animals.

The subject of phosphorus elimination has been studied under many pathological conditions, and especially in hunger, in the cases of Cetti,^a Breihaupt,^b and others.

^a Senator and Müller: Virchow's Archiv. Suppl., 1893, 131 : 2.

^b Müller: Virchow's Archiv, Suppl., 1893, 131 : 52.

A phosphoric-acid diabetes, noted by Teissier,^a and Ralfe,^b showed a resulting polyurea where as much as 12 grams of phosphoric acid were eliminated per day by the kidneys. In diseases of the kidneys the activities of these organs in eliminating the phosphates may be considerably diminished. In meningitis, on the contrary, a marked increase in the phosphates eliminated is observed in the urine. The statements in regard to the quantity of phosphates in the urine in rachitis and in osteomalacia are somewhat contradictory. A phosphaturia is described, which is more correctly called an alkalinuria, where the phosphates settle out owing to an alkaline reaction. A pathological phosphaturia is also noted. Sendtner^c showed that there was an increased calcium excretion in cases of phosphaturia. This condition is due to a perversion of metabolism, but serves to illustrate the close relationship which exists between calcium and phosphoric acid.

Voit^d found that the feces of starving dogs contained phosphates.

The subject of phosphorus elimination has been quite fully investigated by Paton, Dunlop, and Aitchison.^e In the case of dogs fed on a vegetable diet a large proportion of the phosphorus of the food is not eliminated in the urine. The same thing is true when the phosphorus (inorganic) is injected subcutaneously.

In the case of goats none of the subcutaneously injected phosphorus is found in the urine, neither is any of the body or food phosphorus found in the urine. During lactation the excretion of phosphorus by the bowel is diminished to meet the requirements of milk formation. In the case of dogs there is a diminished excretion of phosphorus in the urine during lactation. The milk of goats contains a large amount of total phosphorus, but a small percentage of organic combined phosphorus.

On giving a soluble glycono-phosphate of calcium by the mouth no increased excretion of phosphorus was detected in the urine of dogs or in the urine or milk of goats.

The excretion of inorganic constituents in the urine was studied by Cathcart and Fawsitt^f during a fourteen-day fasting period. The excretion of phosphorus fell off gradually. There was a decreased output of calcium, magnesium, sodium, and potassium. The normal ratio of sodium and potassium is reversed in starvation.

Fitz, Alsberg, and Henderson's^g determinations of phosphoric acid excretion during experimental acidosis in rabbits are to the

^a *Lyon Médical*, 1875, 19 : 307.

^b *Lancet*, 1887 (2), p. 1243.

^c *Münch. med. Wochenschr.*, 1888, 35 : 671.

^d *Hermann's Handbuch der Physiologie*, 1881, 6 : 345.

^e *J. Physiol.*, 1900, 25 : 212.

^f *Ibid.*, 1907, 36 : 27.

^g *Amer. J. Physiol.*, 1907, 18 : 113.

effect that feeding hydrochloric acid produces first an increase and then a decrease in the phosphorus (P_2O_5) excreted in the urine. The determination favors the view that the body phosphates are concerned with neutralizing the acid and with its removal from the body.

Roos^a on feeding thyroids to dogs got an increased phosphoric acid excretion and after extirpation of the thyroids found that the elimination of phosphorus was decreased. Phosphorus elimination seems to be regulated, in part at least, by those glands, the relationship being similar to that which probably exists between calcium and phosphoric acid and the ovaries, and that between iron and the spleen.

It is difficult to give a typical urine analysis on account of its variations. The following table may be of some value, though only approximate figures are given for the quantities of the most important inorganic constituents which are eliminated by an average-sized person on a mixed diet in the course of twenty-four hours in a quantity of 1,500 cc:

	Grams.
Sodium chlorid ($NaCl$)	15.0
Sulphuric acid (H_2SO_4)	2.5
Phosphoric acid (P_2O_5)	2.5
Potash (K_2O)	3.3
Ammonia (NH_3)	.7
Magnesia (MgO)	.5
Lime (CaO)	.3
Remaining inorganic bodies	.2
Total	25.0

Phosphoric acid occurs in acid urines partly as double MH_2PO_4 , and partly as simple M_2HPO_4 , both of these phosphates being found in acid urines at the same time. Ott^b found that on an average 60 per cent of the total phosphoric acid was double, and 40 per cent was simple acid phosphate. The total quantity of phosphoric acid is variable and depends on the kind and the quantity of the food. The average quantity of phosphoric acid eliminated by man is in round numbers 2.5 grams, with a variation of from 1 to 5 grams per twenty-four hours. A small part of the phosphoric acid of the urine originates from the burning of organic compounds such as nuclein, protagon, and lecithin within the organism. The greater part originates from the phosphates of the food, and the quantity of eliminated phosphoric acid is greater when the food is rich in alkali phosphates in proportion to the quantity of lime and magnesium phosphates. If the food contains much lime and magnesium, large quantities of earthy phosphates are eliminated in the excrements; and even though the food contains considerable amounts of phosphoric acid in

^a Zts. physiol. Chem., 1895-6, 21: 19.

^b Zts. physiol. Chem., 1886, 10: 1.

these cases, the quantity of phosphoric acid in the urine is small. Such a condition is found in herbivora, whose urine is habitually poor in phosphates. The extent of the elimination of phosphoric acid by the urine depends not only upon the total quantity of phosphorus in the food, but also on the relative amounts of alkaline earths and the alkali salts in the food. According to Preysz^a and Klug^b and Olsavszky, the elimination of phosphoric acid is considerably increased by intense muscular work.

From the transformation of tissues rich in protein or phosphorized nerve substances in the body, an equal relation between the nitrogen and the phosphoric acid in the urine might be expected. Many investigations have been made on this point, but the conditions which affect the elimination of phosphoric acid are not yet sufficiently known to permit any definite conclusions being drawn from the observations thus far made.

Of the various forms of phosphate compounds which appear in the urine the following may be mentioned: Tricalcium phosphate, $\text{Ca}_3(\text{PO}_4)_2$, which occurs only in alkaline urines; calcium diphosphate ($\text{CaHPO}_4 + 2\text{H}_2\text{O}$) occurs in neutral or only in very faintly acid urines; ammonium magnesium phosphate, triple phosphate, may separate, of course, from an amphoteric urine in the presence of a sufficient quantity of ammonium salts, but it is generally characteristic of a urine which has become ammoniacal through alkaline fermentation; amorphous magnesium triphosphate, $\text{Mg}_2(\text{PO}_4)_2$, occurs with calcium triphosphate in urine rendered alkaline by a fixed alkali, and crystalline magnesium phosphate ($\text{Mg}_2(\text{PO}_4)_2 + 22 \text{H}_2\text{O}$) which has been observed in a few cases in human urine, and in horses' urine.

Phosphate calculi may consist of a mixture of the normal phosphate of alkaline earths with triple phosphate. They also are composed of a mixture of earthy phosphate, triple phosphate, and ammonium urate, surrounding a foreign body as a nucleus. Calculi consisting of triple phosphates alone and stones of simple acid calcium phosphate are seldom obtained.

SALTS IN THE ORGANISM.

The body contains in its tissues and liquids a considerable amount of inorganic material. When any organ is incinerated this material remains as ash. If the bones, which are rich in mineral material, are omitted the average amount of ash in the human body amounts to about 0.1 per cent of its weight. It consists of chlorids, phosphates, sulphates, carbonates, fluorids, silicates of potassium, sodium, calcium,

^a Maly's Jahres-Ber., 1891, 21 : 352.

^b Arch. gesam. Physiol., 1893, 54 : 21.

magnesium, and iron; iodine occurs also, especially in the thyroid tissues. In the liquids of the body the main salts are sodium chlorid, sodium carbonate, sodium phosphate, and potassium and calcium chlorid or phosphate. In considering the organic foodstuffs, their value as sources of energy, as well as their function in constructing tissue, is emphasized. The salts have no importance from the former point of view. Whatever chemical changes they undergo are not attended by the liberation of heat energy—none at least of sufficient importance to be considered. They have, however, most important functions, as they maintain a normal composition and osmotic pressure in the liquids and tissues of the body, and by virtue of their osmotic pressure play an important part in controlling the flow of water to and from the tissues. Moreover, these salts constitute an essential part of the composition of living matter. In some way they are bound up in the structure of the living molecule and are necessary to its normal reactions or irritability. Even the proteins of the body liquids contain definite amounts of ash, and if this ash is removed their properties are seriously altered, as is shown by the fact that native proteins when made practically ash-free lose their property of coagulation by heat. The globulins are precipitated from their solutions when the salts are removed. The special importance of the calcium salts in the coagulation of the blood and the curdling of milk is well known, as is also the peculiar part played by the calcium, potassium, and sodium salts in the rhythmical contractions of the heart muscle and the irritability of muscular and nervous tissues. The special importance of the iron salts for the production of hemoglobin is also well known. The nutritive importance of the salts in the diet has been demonstrated by direct experiment.

Dogs were fed by Forster^a upon a diet composed of ash-free fats and carbohydrates, and meats which had been extracted with water until the salts had been reduced. The animals were in a dying condition at the end of twenty-six to thirty-six days. It is probable that they would have lived longer if deprived of food entirely, with the exception of water, since the metabolism of the abundant diet provided aided in increasing the loss of salts from the body. Lunin^b has described experiments which indicate that some at least of our salts must be provided for us in organic combinations such as are found in plant and animal foods. In his experiments he found that mice fared well on a diet of dried cow's milk. If fed, however, on a diet containing the organic but ash-free constituents of milk, namely, sugar, fat, and casein, together with the extracted salts of cow's milk, they died in from twenty to thirty days.

^a Arch. Hygiene, 1884, 2 : 385.

^b Zts. physiol. Chem., 1881, 5 : 31.

In a recent article on alkali salts in the ash of human and cow's milk, Kastle^a states that the practices which have for their object the reduction of the amount of fat in cow's milk, or the addition thereto of mineral matter available for neutralizing the acids resulting from the processes of metabolism are based on sound practical experience, the important difference between the two kinds of milk as to mineral constituents being as follows: (1) Human milk contains relatively more of its mineral matter in utilizable form than cow's milk; (2) it can supply the organism of the child with relatively larger amounts of available alkali in proportion to the protein than cow's milk.

The idea is advanced that in the milk of various animals the inorganic constituents are present in the same proportion as in the ash of the young animals. Bunge^b shows that there is a very close relation between the composition of the ash of young rabbits, dogs, and cats, and that of dog's milk, dog's blood, and dog's blood serum. He also makes the statement that the epithelial cells of the mammary glands select from the blood and give to the milk all the inorganic constituents in the proportion needed by the young animal.

Phosphoric acid is an important constituent of milk. According to the same author woman's milk contains 0.31 to 0.45 gram of phosphoric acid per liter, and cow's milk 1.81 to 1.97 grams. It is an important fact that the food of the young furnishes the phosphoric acid in organic combinations.

Many investigators have shown that the phosphorus of cow's milk is not so well absorbed as that of woman's milk. Stoklasa^c claims that the lecithin phosphorus content of woman's milk is 0.35 per cent, as compared with 0.5 per cent in cow's milk. Blauberg^d fed a child on mother's milk and studied the metabolism of the salts contained therein. He compared his results with the results obtained by other investigators and concluded that the constituents of mother's milk seem to be better utilized by the system than the constituents of cow's milk.

No complete analyses of the mineral substances of pure, blood-free muscle substance were found. The ash remaining after burning the muscle (which amounts to about 10 to 15 parts per thousand, calculated on the moist muscle) is acid in reaction. The chief mineral constituents are potassium and phosphoric acid. Next in amount are sodium and magnesium, and lastly calcium, chlorin, and iron oxid. Sulphates only exist as traces in the muscles, but are formed by the burning of the proteins, and, therefore, occur in abundant quantities in the ash. The muscles contain such large quantities of potassium and

^a Amer. J. Physiol., 1908, 22 : 284.

^b Zts. Biol., 1874, 10 : 111, 295.

^c Zts. physiol. Chem., 1895-6, 21 : 79.

^d Abs., Chem. Centrbl., 1897, 68 : 957.

phosphoric acid that potassium phosphate seems to be unquestionably the predominating salt. Chlorin is found in such insignificant quantities that it is perhaps derived from a contamination with blood or lymph. The quantity of magnesium is about double that of calcium. These two bodies, as well as iron, occur only in very small amounts.

Sherman^a in making a determination of the amount of mineral matter required by the human body, examined twenty American dietaries for ash and compared the amount of mineral matter contained in them with the estimated maintenance requirements as found in metabolism experiments. He concludes that iron and protein run parallel and that calcium and phosphoric acid vary; further that the average diets do not supply a sufficient amount of either calcium or phosphoric acid, and as much attention should be paid to the supply of calcium, phosphoric acid, and iron as to protein. Milk and cheese might be substituted for a part of the meat of the ordinary diet and the use of fruits and vegetables should supply a part of the sugar, starch, and minerals. Several other workers have studied ash-free diets.

CALCIUM COMPOUNDS.

The metabolism of calcium has been extensively studied. There are two forms of calcium which enter into the composition of our food and drink, the organic form in milk, eggs, plant seeds, etc., and the inorganic form, which consists principally of calcium carbonate, calcium sulphate, and calcium phosphate. Both forms are absorbable, the amount absorbed depending on the food taken simultaneously. Among other factors influencing the calcium absorption may be mentioned sodium chlorid, which increases, and the alkalis, which diminish, the amount of calcium absorbed. As noted before, there exists a close relationship between calcium and phosphoric acid.

According to Bunge^b and Bertram^c the calcium in plant food is not so well absorbed as that in animal food. Many foods lack calcium, the daily need of which for the human body is, in the opinion of Oberndörffer,^d 1.5 grams, while Bunge^e claims double that amount, or 3 grams per diem. The young need milk rich in calcium. Bunge says calcium forms 0.04 per cent of the blood, while Albu and Neuberg^f cite experiments showing that calcium forms as much as 0.27 per cent of the blood. In arterio sclerosis, Gazert^g

^a Lake Placid Conf., Home Econ. Proc., 1907, 9 : 114.

^b Lehrbuch der Physiologie des Menschen, 1901, 2 : 88.

^c Abs., Chem. Centrbl., 1879, 10 : 526.

^d Berlin. klin. Wochenschr., 1904, 41 : 1068.

^e Zts. Biol., 1876, 12 : 191.

^f Mineral Stoffwechsel, Berlin, 1906.

^g Deutsch. Arch. klin. Med., 1898, 62 : 390.

found from fifteen to twenty times as much calcium in the blood as in normal health. More calcium is absorbed from natural food than from artificial, and in the latter case more calcium is likely to be excreted by the bowels unaltered than normally. The amount of calcium retained by the tissues and its manner of combination depend both upon the quality of the food and the amount of calcium in it.

With the exception of the importance of the alkaline earths as carbonates, and especially as phosphates, on the physical composition of certain structures, such as the bones and teeth, their physiological importance is nearly unknown. The occurrence of earthy phosphates in all proteins, and their great importance in the passage of the proteins from a soluble to a coagulable state, make it probable that the earthy phosphates play an important part in the organization of the proteins. An insufficient supply of alkali earths in the food raises an interesting question as to the effect of this lack on the bony structure.

CALCIUM SALTS AND COAGULATION.

The property which is the most characteristic of casein is that it coagulates with rennet in the presence of a sufficiently great amount of lime salts. In solutions free from lime salts the casein does not coagulate with rennet, but if lime salts are added it is changed so that the solution (even if the enzym is destroyed by heating) yields a coagulated mass, having the properties of curd.

According to Soxhlet^a the soluble lime salts are only of essential importance in coagulation, while the calcium phosphate is without importance. According to Courant^b the calcium casein-compound on coagulation may carry down with it, if the solution contains dicalcium phosphate, a part of this as tricalcium phosphate, leaving monocalcium phosphate in the solution. The chemical process which takes place in the rennet coagulation has not been thoroughly investigated.

The fibrin ferment, which was called thrombin by Schmidt,^c is produced, according to Pekelharing,^d by the action of soluble calcium salts on a preformed zymogen existing in the noncoagulated plasma. Schmidt admits the presence of such a mother-substance of fibrin ferment in the blood and calls it prothrombin.

Brücke^e showed long ago that fibrin left an ash containing calcium phosphate. The fact that calcium salts may facilitate or even cause a coagulation in liquids poor in fibrin ferment has been known for a

^a Münch. med. Wochenschr., 1893, 40: 61.

^b Arch. gesam. Physiol., 1891, 50: 109.

^c Zur Blutlehre, Leipzig, 1892.

^d Zts. physiol. Chem., 1896-7, 22: 245.

^e Vorlesungen über Physiologie, 2nd ed., 1875, 1: 270.

number of years through the researches of Green,^a Ringer and Sainsbury,^b and others. The necessity of the lime salts for coagulation was first shown positively by the important investigations of Arthus and Pagès.^c In regard to the manner in which the lime salts act a conclusion has been reached by Freund,^d who claims that the separation of the excess of calcium phosphate is the cause of a part of the protein becoming insoluble—that is, a cause for coagulation. Weighty objections to this view can be raised, and it is refuted by Latschenberger^e and Strauch.^f According to Pekelharing^g the process is as follows: The prothrombin is converted into thrombin by the action of the soluble lime salts, and fluids which are in all other respects capable of coagulation, but contain only prothrombin and no thrombin, can therefore be coagulated by the addition of soluble lime salts. Thrombin is a lime combination of prothrombin, and the process of coagulation consists in the thrombin carrying the lime to the fibrinogen, which is converted into the insoluble combination of fibrin and lime. Several important papers have appeared, notably those of Field,^h Morawitz,ⁱ and Loeb,^j dealing with the rôle of calcium in the coagulation of the blood. While the literature on this subject has not been fully covered in this report, its importance demands more than a passing reference in a paper dealing with calcium metabolism. It has been shown by the investigations of Cavazzani^k that the lime salts are of importance in the coagulation of the muscle-plasma as well as in that of the blood.

The inorganic constituents of the bony structure, the so-called bone earths, which remain after the complete calcination of the organic substance as a white, brittle mass, consist chiefly of calcium and phosphoric acid, but also contain carbon dioxide and, in smaller amounts, magnesium, chlorine, and fluorine. Alkali sulphates and iron, which have been found in bone ash, do not seem to belong to the bone tissue itself, but to the nutritive fluid or other parts of the bones. According to Gabriel^l potassium and sodium are essential constituents of bone ash. The opinions of investigators differ somewhat as to the manner

^a J. Physiol., 1887, 8:372.

^b Ibid., 1890, 11:369.

^c Abs., Chem. Centrbl., 1891 (1), p. 511.

^d Ibid., 1889 (1), p. 545.

^e Abs., Chem. Centrbl. 1890 (1), p. 169.

^f Blutgerinnungstheorie. Diss., Dorpat, 1889.

^g Abs. Chem. Centrbl., 1892 (2), p. 335.

^h Centrbl. Physiol., 1903, 17:529.

ⁱ Deut. Arch. klin. Med., 1903-4, 479:1.

^j Beitr. chem. Physiol. Path., 1903-4, 5:191; 1904-5, 6:260; Arch. Path. Anat. Physiol., 1903, 173:35; 1906, 185:160; J. Med. Research, 1903, 10:407.

^k Maly's Jahres-Ber., 1892, 22:346.

^l Zts. physiol. Chem., 1893-4, 18:257.

in which the mineral bodies of the bony structure are combined with each other. Chlorin and fluorin are present in the same form as in apatite ($\text{CaFl}_2, 3\text{Ca}_3\text{P}_2\text{O}_8$). If the magnesium, chlorin, and fluorin be eliminated, the last according to Gabriel occurring only as traces, the remaining mineral bodies form the combination $3(\text{Ca}_3\text{P}_2\text{O}_8)\text{CaCO}_3$. According to this author the simplest expression for the composition of the ash of the teeth is $(\text{Ca}_2(\text{PO}_4)_2 + \text{Ca}_3\text{HPP}_3\text{O}_{13} + \text{H}_2\text{O})$, in which 2 to 3 per cent of the lime is replaced by magnesia, potash, and soda, and 4 to 6 per cent of the phosphoric acid by carbon dioxid, chlorin, and fluorin. Analyses of bone earths have shown that the mineral constituents exist in rather constant proportions, which is nearly the same in different animals. The diverse quantitative composition of the various bones of the skeleton depends probably on the varying quantities of other formations, such as the marrow, blood vessels, etc., which they contain. This probably also explains the larger quantity of organic substance in the spongy parts of the bones as compared with the more compact parts. Schrodtt^a has made comparative analyses of different parts of the skeleton of the same animal (dog), and has found an essential difference. The quantity of water in the fresh bones varies from 138 to 438 parts per thousand. The composition of bones at different ages has not been definitely determined, but according to the analyses made by Voit^b of bones of dogs and by Brubacher^c of the bones of children it appears that the skeleton becomes poorer in water and richer in ash with increase in age. Grafenberger^d has found that the bones of rabbits from $6\frac{1}{2}$ to $7\frac{1}{2}$ years old contained only 14 to 17 per cent of water, while the bones of full grown rabbits from 2 to 4 years old contained 20 to 24 per cent. The bones of old rabbits contain more carbon dioxid and less calcium phosphate than do those of young ones.

CALCIUM METABOLISM.

A great many experiments have been made to determine the change in the bone constituents, for instance, when a ration rich in lime and one deficient in lime is fed, but the results have always been indecisive or contradictory. The attempts to substitute other alkaline earths or clay for the lime of the bones have also given unsatisfactory results. Weiske^e has shown that when young and still rapidly growing rabbits are fed with oats, which are poor in acid and lime, plus magnesium and strontium carbonate, these substances in part pass into the skeleton, but a physiological replacement of lime by magnesium or strontium is not to be expected. On the administration of

^a Maly's Jahres-Ber., 1877, 6 : 207.

^b Zts. Biol., 1880, 16 : 55.

^c Ibid., 1890, 27 : 517.

^d Maly's Jahres-Ber., 1891, 21 : 290.

^e Abs., Chem. Centrbl., 1892 (2), p. 590.

madder the bones of the animal are found to be colored red after a few days or weeks; but these experiments have not led to any positive conclusion in regard to the growth or metabolism of the bones.

Under pathological conditions, as rachitis and softening of the bones, an ossein has been found which does not give any typical gelatin on boiling with water. Otherwise pathological conditions seem to affect chiefly the quantitative composition of the bones, and especially the relationship between the organic and inorganic substances. Attempts have been made to produce rachitis in animals by the use of foods deficient in lime. From experiments on fully developed animals contradictory results have been obtained. In young, undeveloped animals Voit^a produced, by lack of lime salts in the food, a change similar to rachitis. In full-grown animals the bones were changed after a long time because of the lack of the lime salts in the food, but did not become soft, only thinner (osteoporosis). The experiments in which the lime salts were removed from the bones by the addition of lactic acid to the food have led to no positive results (Heitzmann,^b Heiss,^c Baginsky^d). Weiske^e on the contrary, has shown by administering dilute sulphuric acid or monosodium phosphate with the food (presupposing that the food gave no alkaline ash) to sheep and rabbits, that the quantity of mineral matter in the bones might be diminished. A few investigators are of the opinion that in rachitis, as in osteomalacia, a solution of the lime salts by means of lactic acid takes place. This was suggested by the fact that Weber and Schmidt^f found lactic acid in the cyst-like altered bony substance in osteomalacia. Well-known investigators have disputed the possibility of the lime salts being washed from the bones in osteomalacosis by means of lactic acid. The recent investigations of Levy^g contradict the statement as to the solution of lime salts by lactic acid in osteomalacia. He has found that the normal relationship $6\text{P}_2\text{O}_5:10\text{Ca}$ is retained in all parts of the bones in osteomalacia, which would not be the case if the bone earths were dissolved by an acid. The decrease in phosphates occurs in the same quantitative relationship as the carbonate; and, according to Levy, in osteomalacia the exhaustion of the bone takes place by decalcification, in which one molecule of phosphate and calcium after the other is removed.

^a Zts. Biol., 1880, 16 : 55.

^b Maly's Jahres-Ber., 1873, 3 : 229.

^c Zts. Biol., 1876, 12 : 151.

^d Virchow's Archiv, 1882, 87 : 301.

^e Abs., Chem. Centrbl., 1892 (2), p. 590.

^f Cited from Gorup-Besanez. Lehrbuch der physiologischen Chemie, 3d Edition, Braunschweig, 1874, p. 636.

^g Zts. physiol. Chem., 1894, 19 : 239.

The relative amounts of calcium and phosphoric acid in the teeth are, according to the analysis of Hoppe-Seyler,^a about the same as in bone earths.

The importance of calcium for the activity of the nervous system and the muscles has been the subject of study by many investigators. The conclusions drawn are that if the amount of calcium is decreased nervous and muscular irritability will result and, conversely, that an increase of the calcium will diminish the irritability of the nerves and muscles. Ringer proved that the frog's heart can be kept beating for long periods upon a mixture of sodium chlorid, potassium chlorid, and calcium phosphate or chlorid, and he laid especial importance upon the calcium. The calcium ions are present in relatively small quantities in the blood, but they are absolutely necessary to contractility and irritability. When present in quantities above normal or when in proportional excess over the sodium or potassium ions they cause a condition of tonic contraction that has been designated as calcium rigor. The calcium promotes a state of contraction, the sodium and the potassium a state of relaxation.

Tigerstedt in his text-book states that calcium salts favor the contraction of the heart, while potassium salts are important for its relaxation. Calcium favors muscular movements of low forms of animal life—the contractility of both skeletal and smooth muscles. He cites the experiments of Voit,^b who fed pigeons with food containing no calcium, and found that the bones which were used for movements were normal for calcium, while the sternum and skull bones were brittle and even perforated in places.

Falta and Whitney^c showed that after extirpation of a dog's pancreas, the calcium elimination was increased, though the nitrogen, phosphoric acid ratio remained unchanged. The excretion of uric acid in these cases was doubled.

The importance of calcium salts for the growing organisms is discussed by Aron and Sebauer.^d Special attention was given to the calcium content of the bones, brains, nerves, muscles, and blood. Dogs and rabbits were used, half of them being fed on a calcium-poor diet. The young animal requires at least 1.2 per cent of its body weight of calcium; a diet supplying a smaller amount is called a calcium-poor diet. Under such conditions nervous and other disorders follow, a condition like rickets being established after continued feeding of such a diet; in these cases the bones contain more water than is normal, that is, a water-rich bone is developed whose

^a Hammarsten, *Textbook of Physiological Chemistry*, New York, 1908, p. 440.

^b *Zts. Biol.*, 1880, 16:55.

^c *Beitr. chem. Physiol. Path.*, 1908, 11:224.

^d *Biochem. Zts.*, 1908, 8:1.

organic framework is poor in calcium. The calcium content of the flesh and blood shows no variation and the brain but a slight variation from the normal.

Following some experiments made by Sanford and Lusk at the Yale Medical School on new-born pigs, Wilson^a studied the influence of diet on the growth of young pigs. Three pigs were killed and analyzed at birth and three were reared on a skim-milk diet. To the diet of one pig, lactose was added; to that of the second, dextrose; and the third was given the skim milk without any added substance. The lactose-fed pig thrived best, while the pig fed on skim milk alone showed the least progress after sixteen days. The analyses showed that the pig fed on skim milk used 52 per cent of the calcium in the food for growth; the lactose-fed pig used 70 per cent; and the dextrose-fed pig 64 per cent. The calcium content of the bodies of the pigs at the end of the experiment was 8.29, 8.03, and 8.13 per cent, respectively. Calcium storage evidently depends on the development of the animal rather than on any specific influence of the milk constituents. Herter^b found striking retardations in the development of the skeleton of older pigs fed on skim milk for many months, but no evidence of rickets was seen.

W. Camerer, jr.,^c finds that the calcium content of mothers' milk is barely sufficient to cover the needs of the nursing infant if the percentage composition of the five-months-old baby were the same as that of the new-born baby. The percentage of calcium in the new-born pigs above noted averages 9.4 per cent at birth and is 8.15 per cent at the end of two and one-half weeks' feeding. If the pigs fed on lactose and dextrose had contained 9.4 per cent calcium at the end of the two and one-half weeks' test, an almost complete calcium absorption would have taken place.

Patterson^d states that when an animal is deprived of all inorganic salts in its food profound constitutional disturbances, resulting in death, are produced. The salts of the blood must not only be present in sufficient quantity to bring the osmotic pressure of the blood to a constant value, but they must also be present in certain definite ratios. Every living cell of the body must be washed by a fluid containing salts of certain monovalent and divalent metals in an unvarying ratio, otherwise a disturbance in the intracellular ion-proteins (Loeb)^e or colloidal salts (Osborne)^f is produced. Bearing in mind this necessity for a constant ratio between the various salts of the blood, a number of interesting questions are raised by Patterson in regard to the probable effects of depriving an animal, com-

^a Amer. J. Physiol., 1902, 8 : 197.

^d Bio-Chem. J., 1908, 3 : 39.

^b J. Exper. Med., 1898, 3 : 293.

^e Dynamics of Living Matter, New York, 1906.

^c Zts. Biol., 1902, 43 : 1.

^f J. Physiol., 1906, 34 : 84.

pletely or partly, of one particular metal, say calcium. If the proper ratios are not maintained in the blood, then:

(a) Is the excretion of calcium checked wholly or partially? During the progress of his research an article appeared by Goitein^a which disposes of this question by showing that if a rabbit received less than 0.16 grams of calcium per kilo per day in its food, there was a steady loss of calcium from the body. Lehmann^b and others have shown that in starvation the calcium excreted exceeds the amount of this substance present in the drinking water taken.

(b) Are the other salts of the body reduced *pari passu* by increased excretion? This would entail a considerable fall in the total molecular concentration of the blood, and as the living cells of the body and also the red corpuscles are extremely sensitive to osmotic changes this question may also be answered in the negative.

(c) Is the deficiency in the food made good by certain tissues of the body giving up a portion of their calcium to the blood and so keeping the proper inorganic balance in this fluid? That this would be the most probable contingency may be inferred from a number of facts. Forster,^c who was the first to make observations on the effect of insufficient calcium in the food, found that the muscles lost 56 per cent of their calcium content, while the bones also showed a considerable diminution. Voit^d found that on a calcium-poor diet the bones were more brittle, the skeleton showed a smaller percentage of dry weight than in the normal animal, and that the quantity of calcium in all organs of the body was more or less diminished.

In the experiments in which rabbits were fed on oatmeal and maize meal, a diet which admittedly leads to calcium starvation, the ratio of the calcium of the blood to the total ash of the blood remained the same as that found in the normal animal. That is to say, the blood underwent no loss of calcium relative to the other salts in the time allotted to the experiment—a result which one might anticipate from the immense importance of the salt ratios of the blood. The ratio of calcium to the total mineral matter in the bones was, however, inconstant, and showed fairly wide fluctuations even in the normal animal. The bones can, without doubt, act as store-houses of calcium and possibly of magnesium. That they lose calcium when the animal is placed on a calcium-poor diet has been proved conclusively. Voit's results, however, tend to show that the bones can lose calcium relatively to the other salts, that is, by a selective autolysis. The experiments on his own body metabolism show that calcium can be readily stored during nitrogen retention.

^a Arch. gesam. Physiol., 1906, 115:118.

^b Abs., Maly's Jahres-Ber., 1894, 23:497.

^c Maly's Jahres-Ber., 1873, 3:251.

^d Zts. Biol., 1880, 16:55.

More interesting, however, are the experiments involving rectal feeding, calcium being stored despite a continuous drainage of nitrogen from the body. In the latter case, as the protein absorbed from the food was insufficient, the muscles and glands must have diminished in bulk, and yet calcium was retained. This fact rather points to the bones as the place where calcium is stored. In the experiments on himself, and in those with rectal feeding, with a fixed diet the urinary calcium varied but slightly, and the variations, such as there were, ran parallel with the total amounts of urine excreted. This result is not remarkable if it is assumed that the kidney, in order to lighten its work against osmotic pressure, allows a fraction of each of the salts of the blood to escape into the urine. The greater the volume of the urine, therefore, the greater the amount of salts eliminated.

The following theories have been published by Albu and Neuberg^a concerning the cause of rickets:

1. An insufficient amount of calcium in the food.
2. An inadequate absorption of the calcium salts of the food.
3. A disturbance of calcium retention in the bone-building tissues.
4. A disturbance of calcium absorption in bones themselves (Pfaundler).^b
5. A connection between rickets and blood pressure based on the theory (Stöltzner)^c that calcium metabolism is regulated by a secretion of the kidney.

Similar theories as to the cause of osteomalacia were enumerated by the same author as follows:

1. A lack of calcium in the food.
2. A lack of calcium absorption from the food.
3. A decreased alkalinity of blood, following an excess of free acids in the blood which dissolve the calcium salts of the bone.
4. A perversion of metabolism, (Fehling),^d resulting from a diminished activity of the ovaries, which in time affects the calcium balance.
5. Hoennicke^e classes osteomalacia as a metabolism disease, the phosphorus metabolism being also affected.

In pathological cases the results and opinions are many and diverse in regard to calcium elimination. For example, Beneke^f found increased calcium elimination in fever, while Senator^g obtained opposite results. In characteristic bone diseases, osteomalacia and rickets, the same state of affairs is found.

Calcium and magnesium occur in the urine for the most part as phosphates. The quantity of earthy phosphates eliminated daily is

^a Mineralstoffwechsel, Berlin, 1906.

^b Münch. med. Wochenschr., 1903, 50 : 1577.

^c Jahresbuch f. Kinderheilkunde, 1900, 51 : 73.

^d Arch. Gynaek., 1890-1, 39 : 171; 1894-5, 48 : 472.

^e Berlin. klin. Wochenschr., 1904, 41 : 1154.

^f Grundlinien der Pathologie des Stoffwechsels, Berlin, 1874.

^g Centrbl. med. Wissensch., 1877, 15 : 357.

somewhat more than 1 gram, and of this amount two-thirds is magnesium and one-third calcium phosphate. In acid urines the simple as well as the double acid earthy phosphates are found, and the solubility of the former (among which the calcium salt, CaHPO_4 , is especially insoluble) is particularly augmented by the presence of double acid alkali phosphates and sodium chlorid in the urine (Ott).^a The quantity of alkaline earths in the urine depends upon the composition of the food.

MAGNESIUM COMPOUNDS.

The relative ratio of magnesium to calcium as eliminated by the body is 1 : 8 or 1 : 9, and consists largely of magnesium phosphate, $\text{Mg}_3(\text{PO}_4)_2$. The amount of magnesium required by the body per day is 0.6 gram. As in the case of iron, though magnesium is necessary to health, but little magnesium is found in the child's food, namely, milk. The need of magnesium in the system has been studied by Bunge.^b The magnesium balances have been studied by Blauberg,^c Cronheim and Müller,^d Bertram,^e and Renvall,^f but are not considered as important as the calcium. Moreover, little study has been given to the elimination of magnesium under pathological conditions.

The elimination of phosphoric acid, calcium, and magnesium depends principally on the character of the food and the relative proportion of animal and vegetable food digested.

A FEEDING EXPERIMENT WITH RABBITS.

PLAN OF THE EXPERIMENT.

In these experiments four female rabbits were used, the diet containing as little phosphorus as possible. To two of the rabbits organic phosphorus in the form of crude phytin was fed, and to the other two an equivalent amount of phosphorus in the form of sodium phosphates was given.

It was intended to keep these four rabbits on their respective diets for three or four months, in order that they might become accustomed to the added phosphorus and, further, that it might be completely anabolized, and then to mate them and feed the young rabbits on the same kind of food and on phosphorus in the same respective combinations as that fed to the mother rabbits. When the young

^a Zts. physiol. Chem., 1886, 10 : 1.

^b Zts. Biol., 1874, 40 : 111, 295.

^c Ibid., 1900, 40 : 1.

^d Zts. diät. physik. Therapie, 1902-3, 6 : 25, 92.

^e Abs., Chem. Centrbl., 1879, 10 : 526.

^f Skand. Arch. Physiol., 1904, 16 : 94.

rabbits had lived for several weeks on these diets, it was planned to kill them and to examine their bodies in minutest detail for various combinations of nitrogen and phosphorus. The same procedure was to be carried out in the case of the four female rabbits, and in addition, normal rabbits were to be examined as controls. Unfortunately, it proved impossible to obtain young rabbits under these abnormal conditions, that is, living in closely confined quarters (cages) and fed on an artificial diet.

The work was begun early in November, 1907, and concluded the middle of March, 1908. Complete nitrogen^a and phosphorus balances were determined during a period of nearly five months. Moreover, the inorganic phosphorus was estimated in the urine by the uranium acetate method throughout the entire time. In addition, during the last four weeks, calcium, magnesium, and ether-alcohol soluble phosphorus (lecithin) balances were included to make the study of the phosphorus metabolism more complete.

At the end of the period the rabbits were chloroformed, and the bones, teeth, blood, livers, nerves (including the spinal cord) and brains were analyzed for nitrogen, total phosphoric acid, lecithin-phosphoric acid, calcium, magnesium, water, ash, and ether extract. Two normal female rabbits were chloroformed and the same procedure followed as in the case of the rabbits artificially fed. In all cases post-mortem examinations were made and slides of the various tissues were prepared and histological changes noted.

PREPARATION OF FOOD.

The food consisted of carrots, gluten, a mixture of starch and sugar, olive oil, and salt solution. The above constituents seemed to furnish a well-rounded ration, supplying sufficient protein, fat, and carbohydrate for the needs of the body. The rabbits to which the inorganic phosphorus salts were fed received daily 5 cc of a standard salt mixture consisting of 450 grams of sugar, 4 grams of calcium chlorid, 15 grams of sodium chlorid, 30 grams of potassium chlorid, and 1 gram of magnesium sulphate, made up to a volume of 2,000 cc and containing 0.0492 gram of phosphoric acid, in the form of disodium hydrogen phosphate and sodium dihydrogen phosphate, per cubic centimeter.

The rabbits to which the organic phosphorus was fed received daily 5 cc of a salt mixture made so as to supply an equivalent amount of the above mineral salts, allowance being made for the presence of calcium, magnesium, potassium, and phosphorus in the phytin. In this way an equal amount of calcium, magnesium, potassium, and

^a All the nitrogen work was done by the nitrogen laboratory, Mr. T. C. Treacot in charge.

sodium was given to all four rabbits, and the total amount of phosphoric acid fed was practically equalized.

Gluten was selected as a food high in nitrogen but containing little phosphoric acid. The usual method of washing out the starch from coarse flour was employed. The moist gluten was spread out on sheets of tin and dried on the steam bath. After several days of this treatment the samples were sufficiently dried for grinding, and contained from 12 to 13 per cent of nitrogen.

The organic phosphorus was supplied in the form of phytin, a calcium-magnesium-potassium compound of anhydro-oxy-methylene-di-phosphoric acid which was first isolated by Posternak.^a This was prepared by extracting wheat bran with 0.2 per cent hydrochloric acid, allowing the starch to settle, decanting off the clear liquid, and to this adding a large volume of 95 per cent alcohol. A heavy flocculent precipitate formed. This was allowed to settle and after the clear liquid had been decanted off, the remainder was filtered. The precipitate was then dried at room temperature by blowing air over it by means of an electric fan. In this air-dried condition the phytin contained from 22 to 30 per cent of phosphorus (P_2O_5) in organic form. The uranium acetate titration method showed that no inorganic phosphorus was present.

The nature of phytin has been investigated by Patten and Hart,^b who gave to it the following composition: Calcium, 1.13; magnesium, 5.80; and phosphorus, 16.3 per cent.

Phytin on heating with mineral acids is decomposed into inosite and phosphoric acid. The investigators just quoted claim there is no decomposition of phytin by enzymes and the same conclusion was reached by Mendel and Underhill,^c who also studied this question. It is claimed that the proteolytic enzymes of the alimentary tract do not alter phytin, but that the alteration is brought about by the intestinal epithelium. The free acid phytin corresponds to the formula $C_2H_4P_2O_6$. The alkali salts are freely soluble in water and the calcium and copper salts are slightly soluble in water, while the barium and strontium salts are but sparingly soluble in water. Phytin has thus far been found in peas, beans, pumpkin seeds, and red and yellow lupines. The carbohydrates of the food were supplied by feeding a mixture consisting of equal portions of cane sugar and cornstarch. The fat used was olive oil.

The food was prepared in the following manner: The carrots were first chopped into small pieces and a portion was mixed with part of the gluten-starch-sugar mixture. To this was added the phytin and 5 cc of the phosphoric-acid-free salt solution in the case of the rabbits

^a Rev. gen. bot., 1900, 12 : 5.

^b Amer. Chem. J., 1904, 31 : 564.

^c Amer. J. Physiol., 1906, 17 : 75.

fed organic phosphorus. This was made into a thick paste and placed on a small tray in one corner of the cage. The same procedure was followed in the case of the rabbits fed inorganic phosphorus, except that instead of phytin the salt solution containing the inorganic phosphorus was mixed with the food. Thus the rabbits were compelled to eat the food containing the phosphorus before the remainder of the food was given to them. The rest of the carrots, sugar and starch mixture, and 2 cc of olive oil were made into a thick paste and given to the rabbits during the afternoon.

METHODS OF ANALYSIS.

Each rabbit was confined in a suitable wire cage, which allowed the feces and urine to be easily separated.

After establishing a nitrogen equilibrium, the experiments with the rabbits were commenced. The feces were collected at frequent intervals during the day, owing to the fact that the rabbits persistently ate them. The urine and the food residues were collected daily. All of these samples were composited and analyzed; the nitrogen in the food, feces, and urine being determined according to the Gunning^a method and the phosphoric acid in the food, feces, and urine by Neumann's^b method. The phosphoric acid in the urine was determined also by the uranium acetate volumetric method. In this way a check on the amount of phosphoric acid in the urine was obtained, and further, this double determination served as an indication of the presence of organic phosphorus.

The methods employed for water and ash, and for calcium and magnesium^c were those of the Association of Official Agricultural Chemists. From 2 to 3 grams of the foods or feces were ashed; and in the case of the urine, 200 cc were evaporated to dryness in a platinum dish and ashed. The ether-alcohol soluble phosphorus (lecithin) was determined in the following manner:

Transfer one or more grams of the finely ground substance to a 300-cc Erlenmeyer flask; add 30 cc of absolute ether and extract the whole over night. Filter the ether extract through a hardened filter paper into an ordinary Jena flat-bottomed flask of 500-cc capacity and return any particles of the residue found on the filter paper to the Erlenmeyer flask. Then add to the ether extract residue 60 cc of absolute alcohol and boil the solution for three hours, using a reflux condenser. Filter this alcohol extract while hot into the Jena flask containing the ether extract and wash the residue twice with two separate portions of 25 cc each of hot alcohol, adding the washings to the extract. In the combined ether-alcohol filtrate determine the phosphoric acid by the Neumann^b method.

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 107, Rev., p. 7.

^b Zts. physiol. Chem., 1902, 37 : 115.

^c U. S. Dept. Agr., Bureau of Chemistry, Bul. 107, Rev., p. 15, 16.

PRELIMINARY FEEDING PERIOD.

The results recorded in Table I are especially valuable in view of the fact that they cover a period of practically one hundred days. During this time an attempt was made to mate the rabbits, each one being removed from her cage every four days at 5 o'clock and placed in a large box with a male rabbit and allowed to remain there until the next morning at 9 o'clock. This was repeated fifteen or twenty times in each case. Consequently some loss of feces and urine must have resulted, which loss in the course of an experiment extending over one hundred days would be practically uniform in the case of each pair of rabbits. Such a loss would naturally tend to give somewhat larger apparent nitrogen and phosphoric acid balances. The rabbits had always eaten the food provided for them before being removed from their cages.

Throughout this work the rabbits fed organic phosphorus are given the numbers 1 and 2, and those fed inorganic phosphorus the numbers 3 and 4.

The weight of the rabbits remained constant in the case of Nos. 1 and 4, while in the case of No. 2 a slight average loss of weight resulted and No. 3 showed a gain in weight.

TABLE I.—*Nitrogen and phosphorus metabolism—Preliminary period.*

No. 1.—RABBIT FED ORGANIC PHOSPHORUS.

Date.	Average weight of rabbit.	Nitrogen (N).				Phosphoric acid (P_2O_5).				In urine.		Absorbed material retained.	
		Total ingested.	Total excreted.		Daily balance.	Total ingested ^a	Excreted.		Daily balance.	Nitrogen.	Phosphoric acid.	Nitrogen.	Phosphoric acid.
			In urine.	In feces.			In urine.	In feces.					
	Gms.	Gms.	Gms.	Gms.	Gms.	Gms.	Gms.	Gms.	Gms.	P. ct.	P. ct.	P. ct.	P. ct.
1907-8.													
November 17-23.....	1,647	3.34	4.22	0.40	-0.18	0.66	0.95	0.28	-0.06				
November 24-30.....	1,596	5.35	4.65	.92	.03	1.81	.13	.90	.11				
December 1-7.....	1,596	8.22	4.91	.83	.35	2.37	.77	.76	.11				
December 8-14.....	1,654	9.33	5.22	.39	.53	3.13	.50	.49	.30				
Average.....	1,623	6.56	4.75	.64	.17	1.99	.59	.61	.11	72.40	29.65		
December 15-21.....	1,656	10.10	4.78	.60	.68	3.29	.68	.69	.27				
December 22-28.....	1,603	10.10	6.33	.60	.45	3.29	1.00	.69	.22				
December 29-January 4.....	1,677	3.54	5.26	.85	.36	1.74	1.12	.77	.02				
January 5-11.....	1,623	7.44	5.12	.54	.25	2.79	.81	.44	.21				
January 12-18.....	1,616	7.03	4.58	1.12	.17	2.67	.63	1.95	.14				
Average.....	1,647	7.64	5.21	.74	.24	2.76	.85	.91	.16	68.19	30.79		
January 19-25.....	1,597	9.56	6.40	1.08	.29	3.27	.77	1.01	.21				
January 26-February 1.....	1,587	10.06	6.06	1.32	.38	3.24	1.04	1.05	.16				
February 2-8.....	1,588	10.19	7.34	1.02	.26	3.27	1.11	1.02	.16				
February 9-15.....	1,567	10.60	8.11	1.33	.16	3.23	1.80	1.16	.03				
Average.....	1,585	10.10	6.98	1.19	.27	3.25	1.18	1.06	.14	69.11	34.71		
Average for period.....	1,618	8.08	5.65	.86	.23	2.67	.87	.86	.14	69.90	31.72	22	53

^a 1.42 grams of organic phosphorus per period were intimately mixed with the food.

TABLE 1.—Nitrogen and phosphorus metabolism—Preliminary period—Continued.

No. 2.—RABBIT FED ORGANIC PHOSPHORUS.

Date.	Average weight of rabbit.	Nitrogen (N).				Phosphoric acid (P ₂ O ₅).				In urine.		Absorbed material retained.	
		Total ingested.	Total excreted.		Daily balance.	Total ingested. ^a	Excreted.		Daily balance.	Nitrogen.	Phosphoric acid.	Nitrogen.	Phosphoric acid.
			In urine.	In feces.			In urine.	In feces.					
1907-8.	Gms.	Gms.	Gms.	Gms.	Gms.	Gms.	Gms.	Gms.	Gms.	P. ct.	P. ct.	P. ct.	P. ct.
November 17-23.	1,816	4.16	4.86	0.51	0.17	1.35	0.82	0.45	0.11				
November 24-30.	1,728	4.32	6.09	.56	.33	1.98	.39	.41	.10				
December 1-7.	1,684	9.97	4.96	.76	.60	2.88	.65	.72	.21				
December 8-14.	1,618	9.95	6.78	1.75	.20	3.26	.66	1.35	.17				
Average.	1,712	7.10	5.67	.90	.16	2.37	.63	.86	.14	79.86	26.58		
December 15-21.	1,542	11.76	7.82	2.26	.23	3.37	.93	1.88	.05				
December 22-28.	1,490	11.76	7.84	2.26	.23	3.37	.88	1.88	.08				
December 29-January 4.	1,505	10.67	5.85	1.19	.51	3.36	.31	.95	.29				
January 5-11.	1,567	10.67	5.40	1.58	.52	3.36	.62	1.60	.16				
January 12-18.	1,646	10.67	5.61	.57	.64	3.36	.41	.54	.34				
Average.	1,548	11.11	6.50	1.57	.43	3.36	.63	1.37	.18	58.51	18.75		
January 19-25.	1,658	11.12	5.55	1.29	.60	3.33	.83	1.24	.18				
January 26-February 1.	1,698	11.71	6.48	1.51	.44	3.31	1.18	1.01	.15				
February 2-8.	1,697	9.11	7.75	2.84	.21	2.63	1.76	1.26	.05				
February 9-15.	1,647	8.85	6.65	.83	.19	2.90	1.20	.65	.14				
Average.	1,675	10.20	6.61	1.62	.36	3.04	1.24	1.04	.11	64.80	40.79		
Average for period.	1,645	9.47	6.26	1.39	.32	2.92	.83	1.09	.14	67.72	28.71	24	52

No. 3.—RABBIT FED INORGANIC PHOSPHORUS.^b

November 17-23.	1,474	7.53	6.48	1.20	-0.02	2.83	0.58	1.02	0.16				
November 24-30.	1,481	9.10	7.46	2.15	-.07	3.27	.96	1.38	.13				
December 1-7.	1,447	10.40	8.89	1.12	.05	3.32	.98	1.38	.13				
December 8-14.	1,436	10.05	7.20	1.15	.24	3.59	.99	1.15	.20				
Average.	1,460	9.27	7.51	1.41	.05	3.25	.88	1.24	.16	81.01	27.08		
December 15-21.	1,401	11.70	5.48	.65	.79	3.67	.84	.25	.36				
December 22-28.	1,484	11.70	6.14	.65	.70	3.67	1.54	.25	.26				
December 29-January 4.	1,526	10.62	4.73	1.66	.60	3.66	.63	.99	.30				
January 5-11.	1,598	10.62	6.50	1.50	.37	3.66	1.08	1.27	.18				
January 12-18.	1,652	10.62	4.89	.88	.69	3.66	.82	.88	.27				
Average.	1,532	11.05	5.55	1.07	.63	3.66	.98	.73	.27	50.23	26.78		
January 19-25.	1,729	11.06	5.82	1.18	.57	3.64	1.06	1.17	.20				
January 26-February 1.	1,766	11.65	5.72	1.62	.61	3.61	1.16	1.58	.12				
February 2-8.	1,776	11.65	6.51	2.54	.37	3.61	1.28	1.97	.04				
February 9-15.	1,778	12.08	7.65	2.54	.26	3.65	.85	1.98	.11				
Average.	1,762	11.61	6.43	1.97	.45	3.63	1.09	1.67	.12	55.38	30.02		
Average for period.	1,585	10.64	6.50	1.48	.38	3.51	.98	1.37	.18	62.21	27.96	29	54

^a 1.42 grams of organic phosphorus per period were intimately mixed with the food.^b 1.72 grams of inorganic phosphorus per period were intimately mixed with the food.

TABLE I.—*Nitrogen and phosphorus metabolism—Preliminary period—Continued.*

No. 4.—RABBIT FED INORGANIC PHOSPHORUS.

Date.	Average weight of rabbit.	Nitrogen (N).				Phosphoric acid (P_2O_5).				In urine.		Absorbed material retained.	
		Total ingested.	Total excreted.		Daily balance.	Total ingested. ^a	Excreted.		Daily balance.	Nitrogen.	Phosphoric acid.	Nitrogen.	Phosphoric acid.
			In urine.	In feces.			In urine.	In feces.					
1907-8.	Gms.	Gms.	Gms.	Gms.	Gms.	Gms.	Gms.	Gms.	Gms.	P. ct.	P. ct.	P. ct.	P. ct.
November 17-23.....	1,950	8.90	8.89	0.76	-0.10	3.10	1.54	0.49	0.15				
November 24-30.....	1,960	9.14	7.91	1.13	.01	3.31	.94	.94	.20				
December 1-7.....	1,960	10.40	4.81	.42	.73	3.32	1.19	.36	.25				
December 8-14.....	1,985	10.05	3.71	.68	.80	3.59	.36	.51	.38				
Average.....	1,964	9.62	6.33	.75	.36	3.33	1.34	.58	.25	65.80	40.24		
December 15-21.....	1,965	11.70	5.16	1.06	.78	3.67	.74	.90	.28				
December 22-28.....	2,002	11.70	8.63	1.06	.32	3.67	1.42	.90	.19				
December 29-January 4.....	1,953	10.62	8.89	1.28	.06	3.66	1.44	1.42	.11				
January 5-11.....	1,952	10.62	6.44	.26	.55	3.66	1.02	.42	.31				
January 12-18.....	2,022	10.62	5.21	.46	.70	3.66	1.01	.57	.29				
Average.....	1,979	11.05	6.86	.82	.48	3.66	1.13	.84	.24	62.08	30.88		
January 19-25.....	2,006	7.78	5.94	1.09	.10	3.20	1.55	1.09	.04				
January 26-February 1.....	2,017	11.65	7.43	.72	.50	3.61	1.29	.77	.22				
February 2-8.....	2,000	7.06	5.44	.46	.16	2.28	1.48	.54	.03				
February 9-15.....	1,975	8.07	6.59	2.38	-.12	1.45	1.03	2.36	-.13				
Average.....	2,000	8.64	6.35	1.16	.16	2.64	1.34	1.19	.04	73.50	50.76		
Average for period.	1,981	9.77	6.51	.91	.33	3.21	1.27	.87	.18	67.12	40.63	.26	.42

^a 1.72 grams of inorganic phosphorus per period were intimately mixed with the food.

NITROGEN BALANCES.

The rabbit which gained in weight, No. 3, received a larger amount of nitrogen than the others. In fact, the amount of nitrogen ingested in all cases varied somewhat, but the total during a period of seven days was from 5 to 6.6 grams of nitrogen per 1,000 grams of body weight, No. 3 receiving the largest relative amount. The average figures show that relatively more nitrogen was excreted in the urine in the cases of rabbits No. 1 and No. 2 (those fed organic phosphorus), than in the cases of No. 3 and No. 4 (fed inorganic phosphorus). The amount of nitrogen eliminated in the feces varied with the individual rabbit. No. 1 eliminated 10.6 per cent and No. 2, 14.6 per cent. No. 3 eliminated 13.8 per cent and No. 4 only 9.3 per cent in this manner. Nos. 1 and 2 retained a smaller proportion of the metabolized nitrogen than did Nos. 3 and 4, the figures being respectively 22, 24, 29, and 26 per cent. This means that the rabbits fed inorganic phosphorus retained a larger proportion of the absorbed nitrogen than did those fed organic phosphorus; and it appears that those fed organic phosphorus excreted in the urine a larger proportion of the ingested nitrogen, but did not utilize this nitrogen so well as did the rabbits fed inorganic phosphorus.

PHOSPHORUS BALANCES.

In all cases, excepting rabbit No. 3, the average amount of phosphoric acid ingested during seven days per 1,000 grams of body weight varied from 1.6 to 1.7 grams; in No. 3 the amount was 2.2 grams. More phosphorus was eliminated by rabbit No. 4 through the kidneys than in any other case. Both of the rabbits fed organic phosphorus and No. 3 fed inorganic phosphorus retained about the same amounts of the absorbed phosphorus (averaging 53 per cent), while the figure for No. 4 is much lower, only 42 per cent. From the average figures, it appears that the rabbits fed organic phosphorus eliminated a smaller percentage of the ingested phosphoric acid in the urine than those fed on inorganic phosphorus. It must be remembered that the amount of phosphoric acid eliminated by the kidneys does not necessarily represent the amount metabolized, for inorganic phosphorus ingested might and undoubtedly does pass through the kidneys without undergoing any change. Of the total phosphoric acid ingested, 32 per cent was found in the feces of No. 1 and 31 per cent in the feces of No. 4, while No. 2 and No. 3 eliminated in this manner 37 and 39 per cent, respectively. Although the rabbits fed inorganic phosphorus excreted a larger amount of phosphorus in the urine than did the others, they retained on the average less of the absorbed phosphorus.

The ratio of nitrogen to phosphoric acid in the food is but slightly above 3:1. This shows a much larger proportion of phosphoric acid than is usually fed in a normal diet. The ratio of nitrogen to phosphoric acid in the urine varies from 5:1 to 7.5:1, being higher in the case of the rabbits fed organic phosphorus, owing to the relatively larger elimination of phosphoric acid in the urine of those fed inorganic phosphorus. This ratio in the feces is rather constant, averaging about 1.1:1 in all cases. The exact ratios are given in Table II.

TABLE II.—*Ratio of nitrogen to phosphoric acid in food and excreta—Preliminary period.*

Rabbit.	Food.	Urine.	Feces.
No. 1.....	3.03:1	6.50:1	1.00:1
No. 2.....	3.24:1	7.54:1	1.27:1
No. 3.....	3.03:1	6.63:1	1.08:1
No. 4.....	3.04:1	5.12:1	0.91:1

PRINCIPAL FEEDING PERIOD.

The principal feeding experiment extended over a period of four weeks, and during this time complete nitrogen, phosphoric acid, calcium, and magnesium balances were determined, as well as the ether-alcohol soluble phosphorus balance. In addition the inorganic phosphorus in the urine was determined by the uranium-acetate titration method. Other salts, as well as the calcium and magnesium salts,

are important for the welfare of the organism, but in phosphorus-feeding experiments the two named stand out most prominently. The second rabbit fed organic phosphorus was in poor condition during the test and died of pneumonia at the end of the second week; consequently the results in this case must not be given the same weight as in the others. Throughout this period all of the rabbits remained practically constant in weight. Nos. 3 and 4 received more nitrogen and phosphoric acid than did Nos. 1 and 2 on an average, but Nos. 1 and 4 received practically the same amounts. The nitrogen and phosphorus balances were positive, except in the case of rabbit No. 2, which died, and naturally would show a negative set of balances.

NITROGEN BALANCES.

The analytical data obtained during the experiment are recorded in Table III.

TABLE III.—*Nitrogen and phosphoric acid balances—Principal period.*

RABBITS FED ORGANIC PHOSPHORUS.

Date.	Rabbit. No.	Total ingested.		Total excreted.				Daily balance.		In urine.		Ab-sorbed material retained.	
		Average weight.	Nitrogen.	Phosphoric acid.		Nitrogen.		Phosphoric acid.		Nitrogen.		Phosphoric acid.	
				In feces.	In urine.	In feces.	In urine.	Nitrogen.	Phosphoric acid.	Nitrogen.	Phosphoric acid.	Nitrogen.	Phosphoric acid.
1908.		Gms.	Gms.	Gms.	Gms.	Gms.	Gms.	Gms.	Gms.	P. ct.	P. ct.	P. ct.	P. ct.
February 17-23.....	1	1,565	9.891	2.824	0.751	6.471	0.602	0.887	0.381	0.191			
February 24-.....													
March 1.....	1	1,551	10.176	3.078	2.449	6.680	1.569	.970	.149	.077			
March 2-8.....	1	1,551	10.745	3.391	1.482	7.560	.998	1.146	.243	.178			
March 9-15.....	1	1,533	10.589	3.483	2.310	6.470	1.461	.489	.253	.218			
Average.....	1	1,550	10.3498	3.194	1.748	6.795	1.157	.876	.256	.166	65.65	27.41	21.057.1
February 17-23.....	2	1,660	7.583	2.626	1.572	7.087	.900	1.307	-.154	.060			
February 24-.....													
March 1.....	2	1,623	8.519	2.451	3.284	7.480	1.738	1.230	-.374	-.085			
Average 2 weeks.....	1,642	8.051	2.539	2.828	7.284	1.319	1.269	-.264	-.013	90.47	49.97		
General average.....	1,596	9.200	2.847	2.288	7.040	1.238	1.073						

RABBITS FED INORGANIC PHOSPHORUS.

February 17-23.....	3	1,765	12.398	3.688	2.378	6.959	1.766	1.345	0.437	0.082			
February 24-.....													
March 1.....	3	1,791	12.414	3.688	2.434	7.429	1.479	1.495	.363	.102			
March 2-8.....	3	1,814	12.149	3.944	2.632	7.882	1.328	1.295	.365	.140			
March 9-15.....	3	1,814	11.962	3.944	1.133	7.882	.534	1.014	.421	.342			
Average.....	1,796	12.231	3.788	2.144	7.308	1.328	1.295	.397	.166	59.75	34.17	27.5	47.3
February 17-23.....	4	1,986	9.157	3.206	.596	7.680	.592	1.800	.126	.116			
February 24-.....													
March 1.....	4	2,028	12.401	3.688	.743	10.211	.440	1.927	.207	.189			
March 2-8.....	4	2,011	12.249	3.933	1.198	7.520	.760	1.540	.505	.219			
March 9-15.....	4	1,971	7.72	3.222	1.212	7.360	.728	1.806	-.114	.098			
Average.....	1,994	10.395	3.487	.937	8.193	.630	1.768	.181	.156	78.82	50.71	13.4	438.1
General average.....	1,895	11.313	3.638	1.521	7.751	.979	1.532	.289	.161	69.29	42.44	20.5	42.7

* Including 1.38 grams of organic phosphorus added to the food per period.

* Including 1.42 grams of inorganic phosphorus added to the food per period.

The amount of nitrogen ingested per 1,000 grams of body weight for a seven-day period was as follows: No. 1, 6.7 grams; No. 3, 6.8 grams; No. 4, 5.2 grams. The amount of nitrogen absorbed per 1,000 grams of body weight per period of seven days was likewise very uniform, excepting for rabbit No. 2, being 5.6 grams for No. 1 and No. 3, 4.8 grams for No. 4, and 3.2 grams for No. 2. The amounts of nitrogen excreted in the urine and feces show considerable variation, the ratio of urine nitrogen to feces nitrogen being highest in No. 4, that is, 8.7:1, and lowest in No. 2, 2.6:1. No. 1 showed a ratio of 3.9:1, and No. 3 a ratio of 3.4:1.

In Table IV the ratios of nitrogen to phosphoric acid, calcium to magnesium, and phosphoric acid to calcium in food, feces, and urine are given. The relation of urine nitrogen to urine phosphorus is highest in the cases of the rabbits fed organic phosphorus. This is due to a larger excretion of phosphorus in the urine of rabbits Nos. 3 and 4, fed on inorganic phosphorus. This ratio in the feces is very regular, being 1.5:1 in three cases and for No. 2 increasing to 2.1:1. A higher ratio of calcium to magnesium is noted in the feces of the rabbits fed inorganic phosphorus. This ratio varies considerably in the urine of the individual rabbits.

TABLE IV.—*Ratios of calcium, magnesium, and phosphorus in food, feces, and urine—Principal period.*

RABBITS FED ORGANIC PHOSPHORUS.

Rabbit No.	In—	N:P ₂ O ₅ .	Ca:Mg.	P ₂ O ₅ :Ca.
1	{ Food.....	3.2	2.6	3.6
	{ Feces.....	1.5	2.7	2.0
	{ Urine.....	7.7	3.8	10.9
2	{ Food.....	3.2	3.0	3.2
	{ Feces.....	2.1	1.9	3.1
	{ Urine.....	5.7	1.0	14.7

RABBITS FED INORGANIC PHOSPHORUS.

3	{ Food.....	3.2	2.9	4.2
	{ Feces.....	1.5	3.5	3.6
	{ Urine.....	5.5	2.2	34.0
4	{ Food.....	2.9	2.9	4.0
	{ Feces.....	1.5	3.7	1.8
	{ Urine.....	4.6	6.6	15.7

The figures show also that the phosphorus-calcium ratios of the food of rabbits Nos. 1 and 2 are lower than in the food of rabbits Nos. 3 and 4; this is due to a larger ingestion of phosphorus in the latter cases. The ratio of phosphoric acid to calcium in the feces varies with the individual case. The phosphoric acid to calcium ratios in the urine again show more phosphorus eliminated by rabbits Nos. 3 and 4 than by rabbits Nos. 1 and 2. The nitrogen and phosphoric acid eliminated in the urine are generally considered to represent the

amounts of the two substances metabolized by the system, but this does not hold in all cases. Rabbit No. 2, for example, which died, shows a much larger amount of katabolized nitrogen and phosphoric acid, as indicated by an increased elimination through the kidneys, but this does not indicate an increased metabolism of these two substances. The ratio of nitrogen to phosphoric acid excreted in the feces shows equal average figures for all the rabbits, excluding the figures for No. 2. If the figures for rabbit No. 2 are included, the average ratio is lower for the rabbits fed inorganic phosphorus. The bulk of the nitrogen (60 to 78 per cent) is eliminated by the kidneys, whereas, on the other hand, only 27 to 50 per cent of the phosphoric acid is thus eliminated. The percentage of absorbed nitrogen which was retained in the system is the same for rabbit No. 1 as for the average of Nos. 3 and 4, receiving inorganic phosphorus.

PHOSPHORUS BALANCES.

The study of phosphoric acid metabolism raises many questions, of which the following are especially important:

- (1) How is the phosphoric acid, ingested in different forms, taken up by the body?
- (2) How is the phosphoric acid changed in the body?
- (3) In what manner is the phosphoric acid eliminated from the body?

Many investigators have attempted to answer some or all of these questions, but no definite answer has been obtained. The generally accepted idea is that the phosphoric acid ingested in different forms is taken up by the body partly in various forms of organic combination and partly, also, in the inorganic or phosphate form. Most of the organic phosphorus taken up by the body—that absorbed from the intestines—is changed to the inorganic or phosphate form, and all such phosphorus is eliminated in the urine as phosphates. This idea that organic phosphorus compounds are more valuable than inorganic combinations of phosphoric acid has been promulgated in the medical literature during the past few years. Nevertheless, many practicing physicians continue to prescribe the inorganic forms, not only of phosphorus, but of iron, calcium, magnesium, etc. Yellow phosphorus is given solely as an alternative.

As the extent of the elimination of phosphoric acid is largely dependent upon the character of the food and the absorption of the phosphates in the intestines, it is apparent that the relationship between the nitrogen and phosphoric acid in the urine can only be approximately constant with a certain uniform food. Thus, on feeding dogs with an exclusive meat diet, as observed by Voit,^a when the

^a Cited by Hammarsten, *A Textbook of Physiological Chemistry*, rev. ed., New York, 1908.

nitrogen and phosphoric acid of the food exactly reappeared in the urine and the feces, the relationship was 8.1:1. In these experiments with rabbits the nitrogen and phosphoric acid ratio in the urine varied from 4.6:1 to 7.7:1. In starvation Wellmann^a has shown that this relationship is changed, namely, relatively more phosphoric acid is eliminated, which seems to indicate that besides flesh and related tissues, also another tissue rich in phosphorus is largely destroyed. The starvation experiments show that this is the bone tissue. Tigerstedt^b claims that only 0.134 gram of phosphoric acid is eliminated in the feces of man daily. For some years it was claimed by many investigators that the elimination of phosphorus and nitrogen should run parallel, both substances being derived from protein, the usual ratio of nitrogen to phosphorus being 7.5:1. In these experiments there is a general tendency in the individual data toward parallelism between the nitrogen and phosphoric acid excretion in the urine, but this ratio is not maintained in the general average, as in the case of the rabbits fed inorganic phosphorus a much larger proportion of the phosphoric acid is absorbed and eliminated by the kidneys. Siven,^c Ehrström,^d and Meyer^e have also shown no parallelism to exist. Phosphorus is used in the formation of the bones and other bodies where no nitrogen is present. Moreover, the ratio of nitrogen and phosphoric acid will sometimes run as low as 3:1.

The amount of phosphoric acid which was fed to all the rabbits was considerably higher than the amount present in their normal diet. In fact, the food itself contained practically a sufficient amount to supply the needs of the system. The result is that by adding an excess of phosphoric acid metabolic changes were induced in all cases to a greater or less extent. The amount of phosphoric acid fed per seven-day period per 1,000 grams of body weight varied from 1.6 grams in rabbit No. 2 to 2.1 grams in the cases of rabbits Nos. 1 and 3. The amount of absorbed phosphorus per 1,000 grams of body weight was practically equal, 1.3 grams, excepting in the case of rabbit No. 2, where the figures show 0.7 gram of phosphoric acid per 1,000 grams of body weight. The ratio of phosphoric acid in the urine to that in the feces shows that the individual element was the most important factor, rabbit No. 4 eliminating a far larger proportional amount by the kidneys than in the case of any other rabbit. The percentage of phosphoric acid eliminated by the kidneys was higher in the rabbits fed inorganic phosphoric acid than in rabbit No. 1, but this simply means that more of the inorganic phosphoric acid passed through the kidneys unaltered, for rabbit No. 1 retained a larger proportion of its absorbed phosphorus than did either No. 3 or No. 4.

^a Arch. gesam. Physiol., 1908, 121:508.

^d Ibid., 1903, 14:82.

^b Skand. Arch. Physiol., 1904, 16:67.

^e Zts. physiol. Chem., 1904-5, 43:1.

^c Ibid., 1901, 11:308.

According to Ehrström,^a who studied phosphorus elimination, from 50 to 88 per cent of the phosphoric acid is eliminated in the urine by the human organism, but the average amount given by different investigators varies from 70 to 80 per cent. With animal food almost all the phosphorus is eliminated in the urine, while with vegetable food a larger proportion of the phosphorus is found in the feces. The amounts of calcium and phosphorus present in the food stand in close relationship to one another. The rabbits eliminated from 27 to 50 per cent of the ingested phosphoric acid in the urine, a considerably lower percentage than in the case of carnivorous animals.

ETHER-ALCOHOL-SOLUBLE PHOSPHORUS BALANCES.

The ether-alcohol soluble phosphorus balances were determined during the principal feeding period. The amount of phosphorus ingested in this form was practically the same in all cases. It is interesting to note the small portion of the 29 per cent of organic combined phosphorus in phytin, which is soluble in ether and alcohol. The figures show that 0.59 per cent of the phosphorus was present in phytin as ether-alcohol soluble phosphorus. This shows that the ether-alcohol extracted phosphorus may represent but a small proportion of the total organic combined phosphorus.

The presence of organic phosphorus in the urine has been discussed, but it is interesting to note that the average figures in Tables V and VI show a slightly larger amount of the so-called organic, or ether-alcohol soluble phosphorus in the urine in the case of the rabbits fed on inorganic phosphorus.

TABLE V.—*Ether-alcohol-soluble phosphoric acid balances—Principal period.*

RABBITS FED ORGANIC PHOSPHORUS.

Date.	Number of rabbit.	Phosphoric acid in- gested.	Excreted as phosphoric acid.			Phosphoric acid bal- ance.	Total phosphoric acid in feces.	Ether-alcohol soluble phosphoric acid of feces in terms of to- tal phosphoric acid.
			In urine.	In feces.	Total.			
1908.								
February 17-23.....	1	Gram. 0.2890	Gram. 0.0180	Gram. 0.0146	Gram. 0.0596	Gram. 0.2380	Gram. 0.6020	Per cent. 2.4
February 24-March 1.....	1	.2923	.0050	.0795	.0845	.2075	1.5680	5.1
March 2-8.....	1	.3081	.0036	.0692	.1028	.2053	.9976	9.9
March 9-15.....	1	.3080	.0000	.1188	.1188	.1893	1.4608	8.1
Average		.2994	.0067	.0848	.0914	.2100	1.1571	6.4
1909.								
February 17-23.....	2	.2413	.0162	.0873	.1035	.1378	.9000	9.3
February 24-March 1.....	2	.2374	.0110	.1925	.2035	.0339	1.7380	26.8
Average.....		.2394	.0136	.1399	.1535	.0859	1.3190	15.1
General average.....		.2694	.0102	.1089	.1225	.1479	1.2381	10.8

^a Skan. Arch. Physiol; 1903, 14: 82.

TABLE V.—*Ether-alcohol-soluble phosphoric acid balances—Principal period—Continued.*

RABBITS FED INORGANIC PHOSPHORUS.

Date.	Number of rabbit.	Phosphoric acid In- gested.	Excreted as phosphoric acid.			Phosphoric acid bal- ance.	Total phosphoric acid in feces.	Ether-alcohol soluble phosphoric acid of feces in terms of to- tal phosphoric acid.
			In urine.	In feces.	Total.			
1908.		Gram.	Gram.	Gram.	Gram.	Gram.	Grams.	Per cent.
February 17-23.....	3	0.2797	0.0133	0.0175	0.0308	0.2489	1.7664	1.0
February 24-March 1.....	3	.2797	.0138	.0232	.0370	.2427	1.4792	1.6
March 2-8.....	3	.2797	.0080	.0248	.0328	.2469	1.5308	1.6
March 9-15.....	3	.2797	.0000	.0090	.0090	.2707	.5340	1.7
Average.....		.2797	.0088	.0187	.0274	.2523	1.3276	1.5
February 17-23.....	4	.2629	.0081	.0386	.0467	.2162	.5920	6.5
February 24-March 1.....	4	.2797	.0336	.0166	.0502	.2295	.4400	3.8
March 2-8.....	4	.2797	.0180	.0930	.1110	.1687	.7600	12.2
March 9-15.....	4	.2797	.0000	.0322	.0322	.2477	.7280	4.4
Average.....		.2755	.0149	.0451	.0600	.2130	.6300	6.7
General average.....		.2776	.0119	.0319	.0437	.2327	.9788	4.1

The figures also show that in the case of the rabbits fed organic phosphorus a considerable amount of ether-alcohol soluble phosphorus is excreted in the feces. The analyses of the feces of the rabbits fed inorganic phosphorus show that there is some ether-alcohol soluble phosphorus always present. This amount must come from the ether-alcohol soluble phosphorus of the food, or from the secretions of the intestinal juices. There is no doubt that the feeding of phytin, which contains 0.59 per cent of phosphorus in this form, greatly increases the amount of ether-alcohol soluble phosphorus in the feces. The insoluble calcium phosphate formed in the gastro-intestinal tract of the rabbits fed inorganic phosphorus may tend to give a higher ratio of ether-alcohol soluble phosphorus in the feces of the rabbits fed with phytin. Further, the results indicate that of the total phosphorus eliminated in the feces the percentage of ether-alcohol soluble phosphorus is much larger in the case of the rabbits fed on organic phosphorus. This may indicate that when phytin is fed in large amounts the gastro-intestinal tract is not able to split and absorb it all. Consequently the percentage of ether-alcohol soluble phosphorus in the feces is increased. The question of the presence of organic phosphorus in the urine is still unsettled. In the principal feeding period the phosphorus in the urine was determined by the uranium acetate titration method, and, further, the lecithin phosphorus, ether-alcohol method, was applied to the urine after evaporating 100 cc to dryness. The figures which are given in Table VI show that the urine results for phosphorus obtained

by the different methods are practically the same whether organic or inorganic phosphorus is fed.

TABLE VI.—*Various forms of phosphorus in urine—Principal period.*

Date.	Number of rabbit.	Phosphoric acid per 100 cc of urine.				Difference between two figures for organic phosphorus.
		Total (by Neumann method).	Inorganic (by uranium acetate method).	Organic (by difference).	Organic (by ether-alcohol extraction).	
1908.						
February 17-23.....	1	Gram. 0.0986	Gram. 0.1009	Gram. -0.0023	Gram. 0.0020	Gram. 0.0043
February 24-March 1.....	1		.1153		.0005	
March 2-8.....	1		.1080		.0003	
March 9-15.....	1		.0670		.0000	
February 17-23.....	2	.1376	.1308	.0068	.0017	.0051
February 24-March 1.....	2	.1230	.1181	.0046	.0011	.0035
February 17-23.....	3	.1416	.1277	.0139	.0014	.0125
February 24-March 1.....	3	.1300	.1287	.0013	.0012	.0001
March 2-8.....	3	.1325	.1290	.0035	.0008	.0027
February 17-23.....	4	.2250	.2039	.0211	.0009	.0202
February 24-March 1.....	4	.1835	.1740	.0095	.0032	.0063
March 2-8.....	4	.1540	.1340	.0200	.0018	.0182
March 9-15.....	4	.1570	.1290	.0280		

An ether-alcohol extraction of an inorganic phosphate solution (100 cc of a microcosmic salt solution) was made containing 0.2 gram of phosphoric acid and this gave 0.0054 gram of phosphoric acid by the ether-alcohol extraction method, as large an amount of ether-alcohol soluble phosphorus as was obtained in the average samples of the urine examined. This fact points to the conclusion that no ether-alcohol soluble phosphorus is normally present in the urine of rabbits, even after the feeding of organic phosphorus for several months.

A review of this question of the presence of organic phosphorus in the urine in general supports this conclusion. Ronald^a was the first to call attention to the presence of organic phosphorus in the urine, and Rockwood^b claims to have found phosphocarnic acid present. Bergmann,^c however, using glycerophosphoric acid made subcutaneous injections on sheep, but could not detect the same in the urine. Patten, Jordan, and Hart^d in their extensive experiments with cows found no organic phosphorus eliminated in the urine, and like results were obtained by Mendel and Underhill^e and by Le Clerc and Cook^f working with rabbits and a dog.

^a Philosophical Transactions, 1864, p. 461.^d Amer. J. Physiol., 1906, 16:268.^b Abs., Chem. Centrbl., 1895 (1), p. 1063.^c Arch. exper. Path. Pharm., 1902, 47:77.^e Ibid., 1906, 17:75.^f J. Biol. Chem., 1906, 2:203.

Mandel and Oertel ^a made some experiments on man along this line, feeding first food poor in phosphorus and later food rich in phosphorus, but found no effect upon the amount of organic phosphorus in the urine. They conclude that the organic phosphorus of the urine is of endogenous origin. The inorganic phosphorus was determined by uranium acetate titration, the solution was boiled with hydrochloric acid and retitrated, the difference being the organic phosphorus.

Organic phosphorus was found in the human urine by Sotnitschewsky.^b Oertel,^c using the method of precipitating the inorganic phosphorus with calcium chlorid in ammoniacal solution, found organic phosphorus in the urine of seven men.

Keller ^d undertook some extensive experiments along this line in the case of infants, and he concluded that the food did not influence the amount of organic phosphorus in the urine. He starved himself and found the amount of organic phosphorus eliminated the first three days was constant, while there was an increased elimination the fourth day. This indicated that some highly organic phosphorized tissue—that is, the lymphocytes—were broken down.

Symmers ^e studied this question in various pathological cases—diseases of the nervous system, enteric fever, tuberculosis, diabetes, and lymphatic leucaemia—and found large amounts of organic phosphorus in the urine.

In agreement with most investigators who have studied phytin and its action on the body, Scofone^f and Giacosa^g claim that phytin is principally eliminated in inorganic combinations.

There is no doubt that in pathological cases there is considerable organic combined phosphorus eliminated, but there is much doubt as to whether any phosphorus in the organic form is eliminated normally in the urine. The methods which have been employed to separate the two forms of phosphorus are far from satisfactory. The slight difference between two results obtained by different methods, which in many cases would be counted as duplicates, has been classed as due to the presence of organic phosphorus. Moreover, in this work, ether-alcohol soluble phosphorus was found in the urine. This is of little significance, because a solution of sodium hydrogen phosphate yielded an equivalent or greater amount of ether-alcohol soluble phosphorus

^a N. Y. Univ. Bul. Med. Sci., 1901, 1 : 165.

^b Zts. physiol. Chem., 1880, 4 : 214.

^c Ibid., 1898-9, 26 : 123.

^d Ibid., 1900, 29 : 146.

^e J. Path. Bact., 1904-5, 10 : 159, 427.

^f Abs., Biochem. Centrbl., 1905, 3 : 606.

^g Ibid., 1905, 4 : 572.

by this same method, which precludes the possibility of any phosphorus in this form being normally present in the urine of rabbits.

In Table VI, where the total phosphorus is determined by the Neumann method and the inorganic phosphorus by titration with uranium acetate, the difference is called organic phosphorus. That the uranium acetate method is not absolutely correct is indicated by the figures which in several cases show more phosphorus by this method than by the Neumann method. In the case of rabbit No. 4 and in one instance in the case of rabbit No. 3 the differences are too large to be explained on the basis of experimental error. It is certain from these results that the ingestion of organic phosphorus does not cause an increased elimination of organic phosphorus in the urine; but the fact that in the case of the rabbits fed inorganic phosphorus there should be an apparent elimination of organic phosphorus in the urine in some cases must be explained on the basis of the endogenous origin of the organic phosphorus of the urine which appears to take place only in abnormal cases.

CALCIUM AND MAGNESIUM BALANCES.

During the principal metabolism experiments, lasting four weeks, the calcium and magnesium balances were determined in addition to those of nitrogen and phosphoric acid. The amount of calcium ingested per seven-day period varied from 0.44 to 0.58 gram per 1,000 grams body weight, while that of magnesium varied from 0.15 to 0.21 gram, rabbit No. 1 receiving more than rabbits Nos. 3 and 4, while rabbit No. 2, which died at the end of the first two weeks, received about the same amount as rabbits No. 3 and No. 4. Goitein ^a states that unless a rabbit receives 0.16 gram of calcium per kilo body weight, a loss of calcium will occur. The figures show that the rabbits under this observation received far more than that minimum amount and, therefore, were in no danger of calcium starvation.

The figures in Table VII show that the calcium excreted in the urine was 9 per cent for No. 1, 10.8 per cent for No. 2, 4.2 per cent for No. 3, and 4.9 per cent for No. 4. In the case of the rabbits fed organic phosphorus, the average amount of calcium absorbed from the intestinal tract or metabolized was higher than in the case of those fed inorganic phosphorus. These figures agree with the theory that the calcium and phosphorus in the inorganic form unite to form the insoluble calcium phosphate, which is eliminated by the bowels in unchanged form.

^a Arch. ges. exp. Physiol., 1936, 115 : 118.

TABLE VII.—Calcium and magnesium balances—Principal period.

RABBITS FED ORGANIC PHOSPHORUS.

Date.	Rabbit No.	Total ingested.		Excreted in urine.		Excreted in feces.		Total excreted.		Daily balance.		Daily balance ratio.		Excreted in urine.		Absorbed material retained.	
		Ca.	Mg.	Ca.	Mg.	Ca.	Mg.	Ca.	Mg.	Ca.	Mg.	Ca.:Mg.		Ca.	Mg.	Ca.	Mg.
1808.		Gm.	Gm.	Gm.	Gm.	Gm.	Gm.	Gm.	Gm.	Gm.	Gm.			P.ct.	P.ct.	P.ct.	P.ct.
February 17-23.	1	0.888	0.330	0.029	0.006	0.407	0.139	0.436	0.145	0.065	0.026	1:0.40					
February 24-March 1.....	1	.863	.320	.166	.053	.771	.257	.937	.310	.011	.002	1:0.18					
March 2-8.....	1	.910	.352	.057	.011	.528	.209	.585	.220	.047	.019	1:0.40					
March 9-15.....	1	.910	.352	.068	.013	.551	.244	.619	.257	.049	.014	1:0.29					
Average.....	1	.893	.339	.080	.021	.564	.212	.644	.233	.037	.015	1:0.32		9.0	6.2	75.6	81.8
February 17-23.	2	.858	.279	.118	.119	.363	.158	.481	.277	.053	.000						
February 24-March 1.....	2	.735	.250	.054	.043	.488	.283	.542	.326	.031	-.011						
Average.....	2	.796	.264	.086	.081	.425	.221	.512	.301	.042	-.006						
General av'ge.....		.844	.301	.083	.051	.494	.216	.578	.267	.040	.005						

RABBITS FED INORGANIC PHOSPHORUS.

February 17-23.	3	0.883	0.307	0.046	0.006	0.669	0.223	0.715	0.229	0.025	0.011	1:0.44					
February 24-March 1.....	3	.893	.307	.029	.046	.281	.100	.310	.146	.083	.023	1:0.29					
March 2-8.....	3	.893	.307	.027	.009	.390	.040	.417	.049	.068	.037	1:0.54					
March 9-15.....	3	.893	.307	.049	.008	.133	.056	.182	.064	.102	.035	1:0.34					
Average.....		.893	.307	.038	.017	.368	.105	.368	.122	.069	.027	1:0.40		4.2	5.5	94.6	91.5
February 17-23.	4	.788	.258	.020	.005	.285	.098	.305	.103	.069	.022	1:0.32					
February 24-March 1.....	4	.893	.307	.050	.015	.261	.063	.311	.078	.083	.033	1:0.40					
March 2-8.....	4	.893	.307	.060	.021	.419	.106	.479	.127	.059	.026	1:0.44					
March 9-15.....	4	.893	.307	.043	.025	.394	.096	.431	.121	.065	.026	1:0.40					
Average.....		.867	.296	.043	.017	.339	.091	.382	.107	.069	.027	1:0.39		4.9	5.7	91.9	91.7
General av'ge.....		.890	.302	.041	.017	.354	.095	.376	.115	.069	.027	1:0.40		4.6	5.6	93.3	91.6

In the case of man, from 5 to 10 per cent of the calcium is normally excreted in the urine; the remainder is excreted with the feces either directly, or a part may be absorbed from the small intestine and excreted into the large intestine, as shown by Voit^a in the case of the dog. Some calcium may come from the intestinal juices. According to Schetelig,^b Von Noorden,^c and Rumpf,^d the amount of calcium excreted in the urine increases with the amount of water taken into the system. Various authors have increased the calcium elimination by adding acids (especially hydrochloric acid) and salts to the food. Hoppe-Seyler^e and Von Noorden found an increased calcium elimination during complete rest. A calcium retention was found by Rumpf, Hirschler, and Terray^f in feeding milk (containing 1.58 grams of calcium per liter). The total volume of urine in each case

^a Cited, Hammarsten, Textbook of Physiological Chemistry, rev. ed., New York, 1908.

^b Virchow's Archiv, 1880, 82:437.

^c Beitr. Lehre Stoffwechsel gesund. krank. Menschen, Berlin, 1902.

^d Berlin. klin. Wochenschr., 1897, 34:265.

^e Zts. physiol. Chem., 1891, 15:161.

^f Zts. klin. Med., 1905, 57:137.

was practically the same, varying only from 975 to 1,100 cc per period on an average, and yet the calcium excreted was subject to considerable variation even in the case of the same rabbit fed on the same diet for several weeks. This fact is contrary to the findings of Patterson,^a who found that on a fixed diet with man the urinary calcium ran parallel to the total amount of urine excreted. Although there was a smaller amount of calcium metabolized by the rabbits fed inorganic phosphorus, yet of this amount a larger proportion was retained than in the case of those fed organic phosphorus.

By excluding the very abnormally high amount of magnesium found in the urine in the case of rabbit No. 2 (which died), we find a very close agreement in the case of the other three animals, though there is a slight tendency for the rabbit fed organic phosphorus to excrete more magnesium in the urine. The amount of metabolized magnesium that was retained shows that the rabbits fed inorganic phosphorus, while metabolizing a smaller amount of the magnesium than did those fed organic phosphorus, retained a larger per cent of the amount actually metabolized. In man from 29 to 38 per cent of the ingested magnesium is excreted in the urine, which is higher than in the case of rabbits. The ratio of calcium to magnesium eliminated in the urine is not constant in the cases studied. According to Bertram^b and Renvall,^c 29 to 38 per cent of magnesium is excreted in the urine and 62 to 71 per cent is eliminated in the feces. It is more easily excreted through the kidneys than is calcium. The ratio of calcium oxid to magnesium oxid excreted in human urine, according to Klemperer and Tritschler,^d varies from 1:0.8 to 1:1.2. The ratio of calcium to magnesium excreted in the feces in the cases of rabbits Nos. 1 and 2 is lower than in the case of Nos. 3 and 4. The ratio of calcium to magnesium excreted by man is held to be 8:1, but in the case of the rabbits the ratio is considerably lower. The amount of magnesium required by man is placed at 0.6 gram per day. In all cases of the rabbits experimented with, daily positive calcium and magnesium balances were obtained.

CHEMICAL ANALYSIS OF THE BODIES OF THE RABBITS.

In all cases the analyses were made on composite samples of two rabbits, and represent the average figures. All analyses were calculated to a water-free basis.

BONES.

The bones were freed from the adhering muscular and tendon tissue and placed in a large kettle and boiled for several hours with water until all the flesh could be easily removed. They were then dried in a hot-air bath, again scraped, and finally ground into a fine powder. The bone powder in the case of the two normal rabbits was

^a Bio-Chem. J., 1908, 3:39.

^b Abs. Chem. Centrbl., 1897, 68:957.

^c Skand. Arch. Physiol., 1904, 16:94

^d Zts. klin. Med., 1902, 44:337.

lighter in color and did not have the oily feeling characteristic of that of the phosphorus-fed rabbits. The water content of the normal bones was lower than in the cases where phosphorus was fed. This was due to the higher fat content in those cases. The remainder of the figures represent the results calculated to a water-free basis, and are shown in Table VIII.

TABLE VIII.—*Chemical analyses of bodies of rabbits (water-free basis).*

RABBITS FED ORGANIC PHOSPHORUS, NOS. 1 AND 2.

Description of sample.	Nitrogen.	Ash.	Calcium.	Magnesium.	Ether extract.	Phosphoric acid.		
						Total.	Ether-alcohol soluble.	Ether-alcohol soluble in terms of total.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Bones.....	4.53	55.56	8.86	0.22	11.33	23.96	0.055	0.23
Livers.....	7.64	3.78	.00	.00	44.95	1.98	.680	34.34
Blood.....	14.72	5.64	Trace.	.00	Trace.	.75	.008	1.07
Brains.....	6.12	7.00	.31	.09	43.23	3.96	2.350	59.34
Nerves.....	5.52	6.54	.21	.11	46.96	3.72	2.390	64.26
Teeth.....		74.65	24.73	1.35		35.31		

RABBITS FED INORGANIC PHOSPHORUS, NOS. 3 AND 4.

Bones.....	4.68	55.92	7.74	0.15	9.39	26.33	0.061	0.23
Livers.....	9.41	4.73	.00	.00	34.48	2.56	.854	33.36
Blood.....	14.42	4.72	Trace.	.00	Trace.	.69	.008	1.16
Brains.....	6.52	7.17	.27	.06	43.89	4.07	1.160	28.51
Nerves.....	3.72	5.83	.28	.06	47.34	4.28	1.470	34.35
Teeth.....		76.10	24.65	1.22		34.70		

NORMALLY FED RABBITS, NOS. 5 AND 6.

Bones.....	4.32	57.47	10.17	0.23	5.12	25.93	0.069	0.27
Livers.....	11.90	5.67	.00	.00	14.47	2.85	1.090	38.24
Blood.....	14.42	4.55	.44	.19	Trace.	.58	.037	6.33
Brains.....	6.85	7.82	.51	.08	38.50	4.16	1.750	42.07
Nerves.....	4.39	5.97	.44	.12	44.34	3.90	2.310	59.23
Teeth.....		75.17	28.35	1.15		35.63		

The normal rabbits show a slightly higher figure for total ash in the bones than do the phosphorus-fed rabbits. Several investigators have pointed out the fact that the skeleton becomes poorer in water and richer in ash with age. Voit^a showed this fact in the case of dogs, and Brubacher^b in the case of children. There is a considerable variation in the ratio of the calcium to the total ash, even in normal animals. Wellmann^c states that in cases of starved rabbits the bones show a smaller percentage of organic matter, that is, a higher ratio of ash. In the case of the normal rabbits, where the highest ash is present, we expect to find the highest percentage of ash constituents, and such is the case.

^a Cited, Hammarsten, Textbook of Physiological Chemistry, rev. ed., New York, 1908.

^b Zts. Biol., 1890, 27 : 517.

^c Arch. gesam. Physiol., 1908, 121 : 508.

The amounts of ether-soluble matter present in the bones show some very interesting results. Both when organic and inorganic phosphorus were fed, the bones contained more ether-soluble matter than the normal bones, evidently a case similar to the increased percentage of ether-soluble material in the liver and presumably due to the phosphorus fed. The bones of the normal rabbits and of those fed inorganic phosphorus show practically an equal amount of total phosphorus, the figure for the rabbits fed organic phosphorus being a little lower. The bones of the normally fed rabbits contain a slightly higher percentage of ether-alcohol soluble phosphorus than in the other cases. The conclusion is that feeding this large amount of phosphorus has not materially affected the quantity of phosphorus stored in the bones, nor has the form of that storage been appreciably changed. There is 0.27 per cent of the total phosphorus as ether-alcohol soluble phosphorus in the bones of the normal rabbits, and 0.23 per cent in the other cases, but the difference, 0.04 per cent, is not large enough to have any significance. In a matter of this kind the age of the animal should be taken into account, for it is known that there is less calcium phosphate and more carbonate in the bones of old rabbits than in the bones of young ones; likewise, less calcium, magnesium, and phosphorus in the bones of starved rabbits, as shown by Wellmann.

On a fat-free, water-free basis the analysis of the bones, given in Table IX, shows a slight variation in the ash content, the ash of the rabbits fed organic phosphorus being slightly higher than that for the bones of the rabbits fed inorganic phosphorus, which in turn is higher than the normal. In regard to the calcium and magnesium, the bones of the rabbits fed inorganic phosphorus contain the smallest amount, while the highest percentage of calcium is present in the normal bones. The total ash content in the cases of the experimental rabbits was greater than in the normal rabbits. The bones of the rabbits fed inorganic phosphorus show a higher phosphorus content than in the other cases; but the results as regards ether-alcohol soluble phosphorus show little variation, the bones of the normal rabbits containing the largest proportion and those of the rabbits fed organic phosphorus the smallest proportion.

TABLE IX.—*Analysis of bones calculated to a fat- and water-free basis.*

Rabbits.	Calcium.	Magnesium.	Ash.	Phosphoric acid.			
				Total.	Ether-alcohol soluble.	Ether-alcohol insoluble.	Ether-alcohol soluble in terms of total.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Fed organic phosphorus.....	9.99	0.25	62.66	27.04	0.065	26.97	0.26
Fed inorganic phosphorus.....	8.54	.17	61.71	29.06	.067	28.99	.23
Normal.....	10.72	.24	60.57	27.33	.073	27.26	.28

LIVERS.

The livers of the rabbits were dried and powdered. The total ash of the livers of the normal rabbits is higher than the ash of the livers of those fed inorganic phosphorus which, in turn, is slightly higher than the ash of the livers of those fed organic phosphorus.

The changes produced in the livers do not seem to include any large storage of phosphorus in the organ, for the livers of the rabbits fed organic phosphorus which show the largest excess of fat contain the lowest percentage of total and ether-alcohol-soluble phosphorus, and the livers of the rabbits fed inorganic phosphorus, which have a lower fat content, contain a lower percentage of phosphorus than the normal rabbits' livers. Not only is there a difference in the total phosphorus content of the various livers, but the livers of the rabbits fed organic phosphorus contain a lower percentage of ether-alcohol-soluble phosphorus than is present in the other cases, the normal livers showing the highest figures. That there is an excess of fat in the livers of the rabbits fed an excessive amount of phosphorus, whether organic or inorganic, is shown by the figures for the ether-soluble matter, namely, 44.95 per cent in the case of the rabbits fed organic phosphorus, 34.48 per cent in the livers of those fed inorganic phosphorus, and only 14.47 per cent in the case of the livers of the normally fed rabbits.

TABLE X.—*Analysis of rabbits' livers (water-free basis).*

No. of rabbit.	Weight of rabbit.	Ration.	Weight of liver.		Percentage composition.			Actual amounts.		Calculated to a fat-free basis.			
			Moist.	Dry.	Nitrogen.	Phosphoric acid.	Ether extract.	Total nitrogen.	Total phosphoric acid.	Nitrogen.		Phosphoric acid.	
										Total.	Per 1,000 grams body weight.	Total.	Per 1,000 grams body weight.
	Gms.		Gms.	Gms.	P. ct.	P. ct.	P. ct.	Gms.	Gm.	Gms.	Gms.	Gms.	Gm.
1.....	1,550	Organic phosphorus	91.0	36.1	7.64	1.98	44.95	2.76	0.72	5.01	3.23	1.31	0.85
3.....	1,800	Inorganic phosphorus	81.5	27.5	9.41	2.56	34.48	2.58	.70	3.94	2.18	1.07	.60
4.....	2,000	do	86.0	35.0	9.41	2.56	34.48	3.29	.90	5.02	2.50	1.37	.69
5.....	2,050	Normal	59.0	14.6	11.90	2.85	14.47	1.74	.42	2.04	1.00	.49	.24
6.....	2,200	do	60.0	15.0	11.90	2.85	14.47	1.79	.43	2.09	.95	.50	.23

In Table X the results for the body weights, liver weights, total nitrogen, total phosphorus, and ether extract found in the livers of the various rabbits are given. That phosphorus in the forms fed tends to enlarge the livers is seen from the percentage of total weight of the livers in terms of the body weight of the rabbits. In the case of Nos.

5 and 6 (the normal rabbits) the livers formed 3 and 2.8 per cent, respectively, of the total weight of the rabbits, while in Nos. 1, 3, and 4 the liver weight made 6.0, 4.5, and 4.4 per cent of the body weight. More total nitrogen and phosphorus is present in the livers of phosphorus-fed rabbits than in the normal livers, and calculated to a fat-free basis the differences are more evident. The tendency of organic phosphorus to produce fat is especially striking; in these experiments an increased amount of ether-soluble matter is seen in the bones, livers, brains, and nerves of the phosphorus-fed rabbits. Jordan, Patten, and Hart,^a in their experiments with phytin, state that when the cows were reduced from a high to a low phytin diet, the percentage of fat in the milk was reduced. The fact that phytin tends to the production of fat in various organs and secretions seems to be established by all of these experiments.

BLOOD.

The figures for blood analysis are given on a water-free basis. The amount of ash is highest in the blood of the rabbits fed organic phosphorus. The blood of those fed inorganic phosphorus and of the normal rabbits contain about an equal amount of ash. The variations are not large enough to be of any consequence when the individuality of the different rabbits is considered. The calcium content of the blood of all the phosphorus-fed rabbits is reduced, showing only a trace, while 0.44 per cent of calcium is present in the dried blood of the normal rabbits. The same holds true in the case of the magnesium, 0.19 per cent being present in the dried blood of the normal rabbits and none being found in the blood of the phosphorus-fed rabbits. The prevailing belief is that the composition of the blood changes but little if any, no matter what changes may take place in the body tissues. Patterson^b found, in the case of animals kept on a calcium-poor diet, that the ratio of the calcium of the blood to the total ash of the blood was the same as that of normal animals. The amount of total phosphorus present in the normal blood is somewhat lower than in the cases where phosphorus was fed. The ether-alcohol soluble (lecithin) phosphorus is highest in the blood of the normal rabbits and the figures show that 6.33 per cent of the total phosphorus is present in the dried blood of the normal rabbits as ether-alcohol soluble phosphorus against 1.07 and 1.16 per cent in the case of the experimental rabbits. Here again we see that the constant ratio of the constituents of the blood is not maintained. Only traces of ether-soluble matter were found in any of the samples of blood examined. (Table VIII.)

^a Amer. J. Physiol., 1906, 16 : 268.

^b Bio-Chem. J., 1908, 3 : 39.

BRAINS.

The brains of the rabbits were completely removed and dried in platinum dishes, then ground in a mortar and mixed as well as possible before analyzing. The ash varied from 7.0 to 7.82 per cent, being highest in the normal brains. As the age and other factors influence the ash content of all organs, this variation has no significance. The percentage of calcium shows some variation and is likewise highest in the ash of the brains of the normal rabbits.

The percentage of magnesium remains fairly constant in all cases. The ratio of calcium to magnesium in the brains is 6.3:1 in the normal rabbits, 4.5:1 in the brains of those fed inorganic phosphorus, and 3.5:1 in the brains of those fed organic phosphorus.

The total phosphorus found in the brains is about the same in all cases, but this is not true of the ether-alcohol soluble phosphorus. The brains of the rabbits fed organic phosphorus show 2.35 per cent phosphorus as ether-alcohol soluble, or 59.34 per cent of the total. The brains of those fed inorganic phosphorus show but 1.16 per cent phosphorus as ether-alcohol soluble phosphorus, or 28.51 per cent of the total. This is lower than the figures for brains of the normal rabbits, which contain 42.07 per cent of the phosphorus in a form soluble in ether and alcohol. It appears from these figures that the brains of the rabbits fed organic phosphorus contain an appreciably larger amount of ether-alcohol soluble phosphorus than do the brains of rabbits fed on inorganic phosphorus or those of the normally fed rabbits. (Table VIII.)

NERVES.

The ash content on a water-free basis shows a fairly constant percentage in all cases. As in the brain, the calcium content of the nerves is higher for the normal rabbits than in the other cases.

The amount of magnesium in the nerves of the normal rabbits is 0.12 per cent, 0.11 per cent, or practically the same, in the nerves of those fed organic phosphorus, and one-half, or 0.06 per cent in the nerves of those fed inorganic phosphorus.

The ratio of the calcium to the magnesium is much lower in the nerves of the rabbits fed organic phosphorus than in the other cases. The data for ether-soluble material are fairly uniform, being a trifle lower for the nerves of the normal rabbits than for the experimental rabbits.

The total phosphorus content varies from 3.72 per cent in the case of the rabbits fed organic phosphorus to 4.28 per cent in the case of those fed inorganic phosphorus. The amount of ether-alcohol soluble phosphorus is lowest in the case of the rabbits fed on inorganic phosphorus, the percentage of the total phosphorus in this form being

34.25 per cent against 64.26 per cent in the case of the rabbits fed on organic phosphorus, and 59.23 per cent in the normal rabbits. These results are similar to those recorded in the case of the brains. (Table VIII.)

TEETH.

The teeth were freed from adhering bone and muscle tissue by scraping and were dried in a hot-air oven at 100° C. The ash was constant in all cases, averaging 75 per cent. There was somewhat more calcium and a trifle less magnesium found in the teeth of the normal rabbits than in the teeth of the phosphorus-fed rabbits. These results run parallel with those obtained for calcium in the case of the bones of the rabbits. The total phosphorus was practically no higher in the normal rabbits than in the other cases. (Table VIII.)

INTESTINES.

Phosphorus was estimated in portions of the small intestines of the various rabbits. The loops of intestine were well washed and dried between filter papers and then in an air bath at 45° C. for five hours. When sufficiently dry they were cut into small bits. Moisture and total phosphorus were determined on a weighed quantity of this substance and on another portion the following extractions were made:

One gram of substance was ground fine with pure sand in a porcelain mortar and transferred to a 300 cc Erlenmeyer flask. Thirty cubic centimeters of absolute ether were then added and the whole extracted on the water bath overnight, using a reflux condenser. The ether extract was then filtered through a hardened filter into an ordinary Jena flat-bottom flask. Particles of residue found on the filter paper were scraped back into the Erlenmeyer flask. To this ether extract residue 60 cc of absolute alcohol were added and boiled for three hours, using a reflux condenser. This alcohol extract was filtered hot into the Jena flask containing the ether extract and the residue washed twice with separate portions of 25 cc of hot alcohol and the washings were added to the combined extract. Phosphorus was determined in the combined ether-alcohol filtrate by the Neumann method.

The ether-alcohol extraction residue was next treated six times with 50 cc portions of cold water saturated with chloroform, allowing ten hours for each extraction, using the same hardened filter paper as before and scraping back the residue from the filter paper into the flask. This solution was evaporated to dryness and the phosphoric acid determined therein by the Neumann method. This is called the phosphorus insoluble in ether and alcohol, but soluble in water. Phosphoric acid was then obtained in the residue by difference and called the phosphorus insoluble in ether, alcohol, and water.

In all cases a large amount of the phosphorus of the fresh sample was soluble in water, fully 50 per cent being dissolved by this means during the preparation of the sample. The amount of phosphorus insoluble in alcohol, ether, and water was higher in the normal rabbits' intestines than in the other cases. The amount of ether-alcohol soluble phosphorus showed but little variation.

SUMMARY.

There are many variations in the composition of the different portions of the rabbits, apparently due to the feeding of phosphorus in the two forms. The amount of ether-alcohol-soluble phosphorus stored in the brains and nerves is much lower in the case of the rabbits fed inorganic phosphorus than in the normal rabbits and those fed organic phosphorus. The organic and the inorganic phosphorus, fed in excess, caused an increased percentage of ether-soluble material in the bones, and an indication of the same is noted in the brains and in the nerves. No increased storage of calcium or magnesium was noted; on the contrary there is a slight decrease, as compared with the normal, in bones, nerves, and brain. The ether-alcohol soluble phosphorus of the blood was reduced in all cases where phosphorus was fed. The increase in the fat of the livers was marked in both cases of phosphorus feeding, but was greater for the rabbits fed organic phosphorus. If a large percentage of ether-alcohol soluble phosphorus in the brains and nerves is desirable, then the rabbits fed organic phosphorus were in a better condition, according to the data, than were those fed on inorganic phosphorus.

The nitrogen content was determined in all of the samples, and is given in Table VIII calculated to a water-free basis. The percentage of nitrogen found in the bones varied from 4.32 per cent in the bones of the normal rabbits to 4.68 per cent in the bones of the rabbits fed on inorganic phosphorus. The percentage of nitrogen found in the livers of the experimental rabbits was considerably lower than in the livers of the normal rabbits, this agreeing with the other data showing a generally poor condition of the livers of the former. The percentage of nitrogen found in the blood was fairly constant, as is usually the case, and the same is true of the brains. Just why we find a variation in the amount of nitrogen present in the nerves and spinal cord is difficult to explain. More ash and more ether-alcohol-soluble phosphorus are found in the samples of nerves of the rabbits fed organic phosphorus, which also show the highest percentage of nitrogen. The ratio of phosphorus to nitrogen in the liver of rabbits is 1:14.7, according to Wellmann.^a His figures are higher than the results obtained in this experiment, the ratio of phosphorus to nitrogen being, for normal rabbits 1:9.7, for rabbits fed organic phosphorus 1:8.8, and for those fed inorganic phosphorus 1:8.6.

^a Arch. gesam. Physiol., 1908, 121:508.

FINDINGS OF AUTOPSIES.

At the conclusion of the principal feeding period the rabbits were chloroformed, autopsies were made, and histological slides of several of the organs prepared. Two normal rabbits were similarly treated. The normal rabbits had been fed on corn, oats, and vegetables for some time previously and had been kept in cages. There seems to be no constant relationship between the total weights and the percentages of solids present in the various organs, blood, brains, and nerves of the six rabbits examined. The autopsies showed the following results: ^a

Rabbit No. 1, fed organic phosphorus.

Intestines: Normal, containing food. Some fat distributed along the intestines.
 Lymphatics: Apparently normal.
 Kidneys: Apparently normal.
 Spleen: Apparently normal.
 Liver: Very light in color, possibly fatty infiltration. Appearance similar to that of No. 2.
 Stomach: Contained food. Normal.
 Heart: Normal.
 Lungs: Showed a condition of anemia along edges.
 After drying: Bones seemed oily. Much fat in liver; fat left in bottom of dish after drying.

Rabbit No. 2, fed organic phosphorus.

Intestines: Normal, except colon distended with large amount of feces; no congestion.
 Lymphatics: Apparently normal.
 Kidneys: Apparently normal.
 Spleen: Apparently normal.
 Liver: Somewhat enlarged, pale, with yellow tinge; seemed pathological.
 Stomach: Full of food; appeared to have extended area of old hemorrhage on lesser curvature.
 Heart: Apparently normal; post-mortem blood clot.
 Lungs: Left, apparently normal; right, partly congested.
 Nervous system: Normal.
 General appearance: Good.

Rabbit No. 3, fed inorganic phosphorus.

Intestines: Apparently normal.
 Lymphatics: Apparently normal.
 Kidneys: There seemed to be slight irritation and congestion.
 Liver: Light colored; enlarged.
 Spleen: Normal in size and color generally; better condition than Nos. 1 and 2; yellow color; numerous small areas of what may be fatty degeneration or infiltration.
 Stomach: Old hemorrhage around greater curvature.
 Heart: Apparently normal.
 Lungs: General appearance good, but apex of right lung had a small area of congestion.
 General appearance: Fairly fat and in good condition externally.

^a H. L. Amoss, of the Animal Physiological Laboratory, assisted in making the autopsies and interpreting the histological slides which were prepared by E. A. Read, of the Microchemical Laboratory.

Rabbit No. 4, fed inorganic phosphorus.

Intestines: Apparently normal.

Kidneys: Right, slight yellow color in cortex, pitted, congested; left, pitted over external surface, slight congestion in medulla, very slight yellowish color in cortex.

Spleen: Apparently normal.

Liver: Light color, lighter spots seen. Gall bladder apparently normal.

Stomach: Capillaries of fundus darkened—apparently not post-mortem change.

Heart: Apparently normal.

Lungs: Left, apparently normal; right, lower half of lower lobe congested.

Rabbits Nos. 5 and 6, normally fed.

The autopsies showed that in the cases of the two normal rabbits, Nos. 5 and 6, the organs were apparently normal. The livers had the normal dark-red color in contrast with the pale livers of Nos. 1, 2, 3, and 4.

The autopsies showed that none of the experimental rabbits was in a normal condition. The livers were especially affected, being pale and abnormal, and indicating an excess of fat.

The relation of the liver to metabolism is a problem important to both the physiologist and the pathologist. For a long time it has been recognized that one of the functions of the liver is connected in some way with the destruction or removal of poison from the blood, especially such poisons as are produced in, or absorbed from, the alimentary canal. It was early recognized that the destruction of the liver cells leads to serious poisoning, and this was experimentally demonstrated by Eck, who excluded the liver from the circulation by making a direct communication between the portal vein and the inferior vena cava, an operation known as "Eck fistula."

The question of fatty degeneration has an important bearing from the physiological point of view, for fatty degeneration is another proof of the formation of fat from proteins. From the investigations of Bauer^a on dogs and Leo^b on frogs we must admit that at least in acute poisoning by phosphorus fatty degeneration takes place with the formation of fats from proteins. Still, investigators are not unanimous on this point, and Pflüger^c especially has raised objections to these experiments.

The ideas of the fatty degeneration of protein in the old sense as used by these writers have been changed by the work of Rosenfeld,^d etc., who believe in the theory of fat transportation.

In the case of a phosphorized dog, Mandel^e has shown that lactic acid disappears from the blood and urine when phlorhizin glycosuria

^a Zts. Biol., 1871, 7 : 63.

^b Zts. physiol. Chem., 1885, 9 : 469.

^c Arch. gesam. Physiol., 1891-92, 51 : 317.

^d Ergeb. Physiol., Pt. I, Biochemie, 1903, 2 : 50.

^e Amer. J. Physiol., Proc., 1905, 13 : 16.

is induced. Lusk^a offers the following general hypothesis as his explanation of fatty changes in tissues:

The lactic acid which occurs is derived from the sugar formed in proteid metabolism. In the above case the sugar is removed before its conversion into lactic acid. In phlorhizin diabetes, dextrose does not burn; in phosphorus poisoning lactic acid derived from dextrose does not burn. In both cases a sugar-hungry cell, or one where carbohydrate is not oxidized, is found, and under these circumstances fat is attracted to the cell, and in larger quantities than can be useful. Wherever sugar freely burns this fatty infiltration is impossible. A reduced local circulation in a portion of the heart may produce anemia of the part, an imperfect local combustion of lactic acid normally formed and a fatty infiltration of the locality.

* * / * * * *

It has been stated that the action of phosphorus is to induce autolysis (self-digestion) of the body's protoplasm (Jacoby,^b Waldvogel^c), since leucin, tyrosin, and other amino acids may be eliminated in considerable quantity in the urine. Oswald^d thinks that phosphorus destroys or weakens the antiautolytic agents of the body. That autolytic enzymes do not gain free control over the cells through the direct influence of phosphorus is proved by the work of Ray, McDermott, and Lusk.^e These authors found that phosphorus injections raised the proteid metabolism of fasting dogs to 250, 260, 283, 248, 183, and 164 per cent of that of the dog when normal. They contrasted this increased proteid metabolism with that obtained in phlorhizin glycosuria, which is represented by increases to 540, 450, 340, and 340 per cent. When, however, they gave phlorhizin and obtained the increased metabolism, and then injected phosphorus, this was not followed by any marked increase in proteid metabolism. Under these circumstances phlorhizin glycosuria is the predominating factor, removing the dextrose produced from proteid. As regards phosphorus poisoning, Araki^f believes that lactic acid accumulation is due to lack of oxygenation of the tissues caused by a slow heart beat, but not due to anemia. He does not believe the oxygen deprivation to be very pronounced. The writer offers the explanation that phosphorus may affect the enzyme which breaks up the lactic acid derived from dextrose, and the accumulation of this acid may prevent the action of some of the denitrogenizing enzymes; and further, its noncombustion may necessitate an increase of proteid metabolism.

This theory is strengthened by the discovery of Schryver^g that the addition of lactic acid favors the accumulation of amino acids in autolysis of the liver.

In this connection we must recognize the fact that the presence of amino bodies—leucin, tyrosin, etc.—in the liver in cases of phosphorus poisoning is well established. Abderhalden and Bergell^h detected glycocoll in the urine of a rabbit poisoned with phosphorus. Wohlgemutⁱ found phenylanin and arginin in the urine after a case of phosphorus poisoning. An altered quantitative relationship between

^a The Elements of the Science of Nutrition, Philadelphia, 1906.

^b Zts. physiol. Chem., 1900, 30 : 174.

^c Arch. klin. Med., 1905, 82 : 437.

^d Biochem. Centrbl., 1905, 3 : 365.

^e Amer. J. Physiol., 1899, 3 : 139.

^f Zts. physiol. Chem., 1893, 17 : 310.

^g Bio-Chem. J., 1906, 1 : 123.

^h Zts. physiol. Chem., 1903, 39 : 464.

ⁱ Ibid., 1905, 44 : 74.

arginin, lysin, and histidin was noted in the liver of a phosphorus-poisoned dog by Wakeman.^a The amount of arginin was considerably lower than the amount present in the liver of a normal dog.

In the experiments here reported there is no doubt that, due to organic and inorganic phosphorus, an alteration in the livers, brains, and nerves has taken place, accompanied by the presence of abnormally high percentages of fat.

HISTOLOGICAL EXAMINATION.

The following results were obtained from the histological examination:

Rabbits Nos. 1 and 2, fed organic phosphorus.

Liver, No. 1: Extensive areas of fatty degeneration and fatty infiltration. Sections stained with hemotoxylin and eosin, Fleming's solution, and Soudan III.

Liver, No. 2: Not quite as extensive areas of fatty degeneration and fatty infiltration as in the case of No. 1. Chronic inflammatory processes seen and general areas of hemorrhages.

Lungs, No. 1: Several areas of focal necrosis noticed surrounding vein in a state of passive congestion. Practically all vesicles collapsed and walls congested. General red cell extravasation.

Stomach, No. 2: Very slight congestion.

Rabbits Nos. 3 and 4, fed inorganic phosphorus.

Liver, No. 3: Infiltration in certain areas, but not as far gone as Nos. 1 and 2. Certain areas showed little change. Slight cloudy swelling. Slight chronic inflammation, not so marked as No. 2.

Liver, No. 4: Little difference compared with Nos. 1 and 2. Some subacute inflammatory processes seen, also some fatty degeneration. Soudan III stain shows more fatty degeneration than is seen with Fleming's.

Lungs, No. 3: Pneumonia in stage of gray hepatization.

Lungs, No. 4: Not normal, but in better general condition than others. Lumen of artery nearly obliterated by fibrous tissue. Bronchitis. Slight general congestion.

Heart, No. 3: Pericarditis. Extensive fatty degeneration of myocardium.

Kidneys, No. 3: Marked congestion. Slight parenchymatous degeneration.

Kidneys, No. 4: Parenchymatous degeneration. Congestion more pronounced than in case of No. 3.

Stomach, No. 3: Apparently normal.

Stomach, No. 4: Apparently normal.

Rabbits Nos. 5 and 6, normally fed.

Livers: Practically normal. No fat present; small areas in which few nuclei did not stain as deeply as the rest, probably due to sectioning.

Lungs: Congestion throughout. A few areas of hemorrhages with superimposed inflammatory processes.

Kidneys: Slight intertubular hemorrhage.

Hearts: Slight myocarditis.

Stomachs: Apparently normal.

The results of the histological examination confirm the opinions formed at the time of the autopsy and show most marked fatty

^a Zts. physiol. Chem., 1905, 44 : 335.

degeneration of the livers of rabbits Nos. 1 and 2. The liver of rabbit No. 4 also shows fatty degeneration, but less marked, while rabbits Nos. 5 and 6 show normal livers. That the harmful effects noted are due to the excessive amounts of phosphorus fed either organic or inorganic, is proven from the cases of the normal rabbits which, although likewise kept in cages, showed apparently normal livers. In reviewing the phosphorus literature it was noticed that in several cases rabbits fed on organic phosphorus were reported to have died of pneumonia, as did rabbit No. 2 in the experiments here recorded.

Six photomicrographs^a are appended, which show sections of the livers of rabbits Nos. 1, 2, and 4. The tissue in the case of rabbit No. 3 was exhausted before an osmic acid slide was prepared and it was therefore impossible to give a reproduction in this case. The photomicrographs in which the fat is shown stained black in position demonstrate that degeneration as well as fatty infiltration has taken place in the livers of both of the organic and in one of the inorganic-phosphorus-fed rabbits.

The author is indebted to Doctor Mohler, of the Bureau of Animal Industry, for his interpretation of the microscopic slides, in regard to which he makes the following statement:

The section in the case of rabbit No. 1 (Pl. I) shows a more marked and advanced lesion of fatty degeneration than do the three remaining cases. In this case fatty infiltration is also present and occurs principally on the periphery of the lobules and in the tract supplied by branches of the portal vein, while the fatty degeneration is more abundant in the central zone around the hepatic vein. In fatty degeneration the fat is usually in minute granules, which may coalesce to form small droplets, but only in the most advanced stages do they form large drops as is the case in fatty infiltration.

The sections in which the tissue has been treated with Fleming's solution show the lesion best, as the contrast of the black-stained fat is so marked as compared with the light hepatic parenchyma that photomicrographs may be readily made. The Sudan III sections, while valuable for demonstration purposes, can not be reproduced as well on account of less contrast and the red color.

The section in the case of rabbit No. 4 (Pl. III) shows a less advanced stage of fatty degeneration; there is also not much fatty infiltration present. In the section in the case of No. 2 (Pl. II) there is still less fatty degeneration present, while the section of rabbit No. 3 does not show any degeneration and only a little fatty infiltration.

CONCLUSIONS.

The somewhat limited experimental data here reported point to the following conclusions, which may be altered by more extensive work on the subject.

PRELIMINARY FEEDING PERIOD.

During the preliminary feeding period, the rabbits fed on organic phosphorus excreted a slightly larger proportion of nitrogen in the urine than did the rabbits fed on inorganic phosphates, but retained

^a Made by B. J. Howard, Chief, Microchemical Laboratory, Bureau of Chemistry.

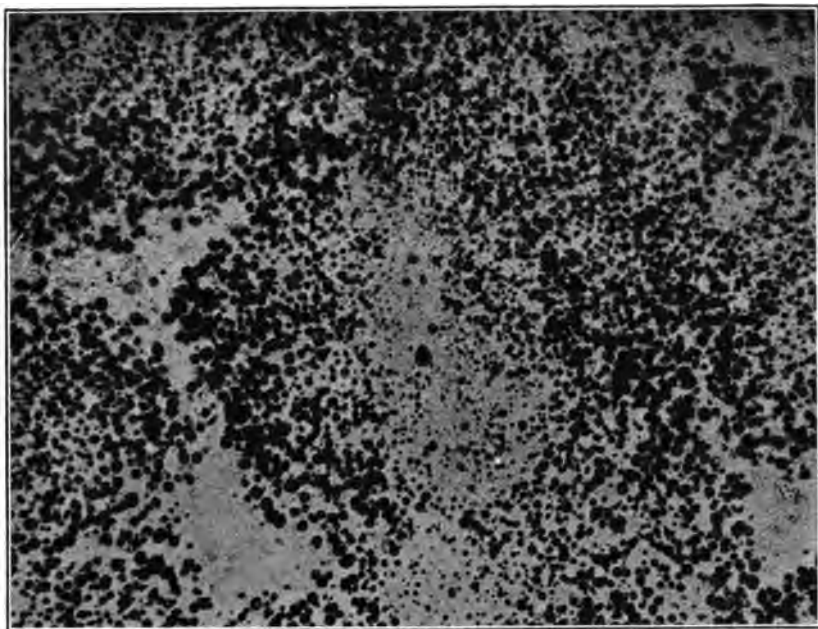


FIG. 1.—MAGNIFICATION 60 DIAMETERS.

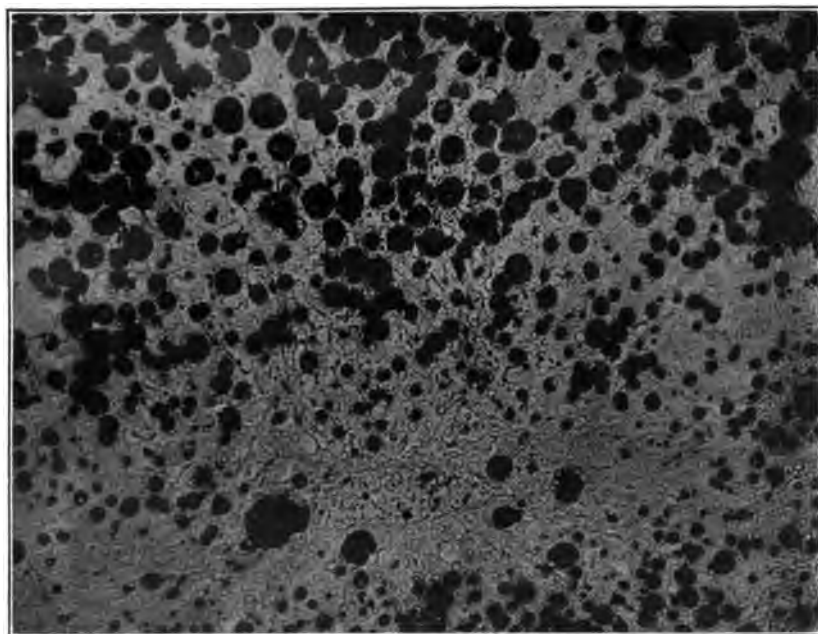


FIG. 2.—MAGNIFICATION 175 DIAMETERS.

LIVER SECTIONS OF ORGANIC-PHOSPHORUS-FED RABBIT NO. 1.

[Fat stained black with osmic acid.]

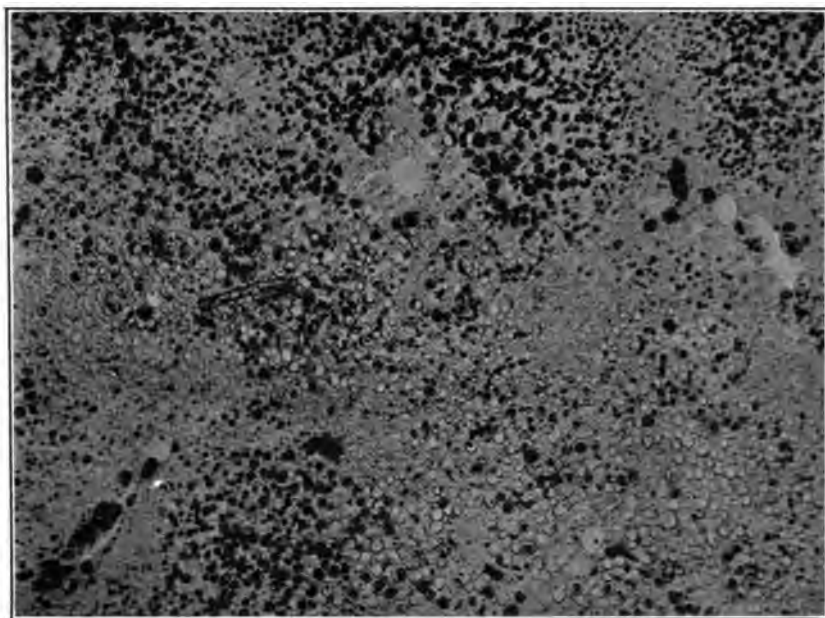


FIG. 1.—MAGNIFICATION 60 DIAMETERS.

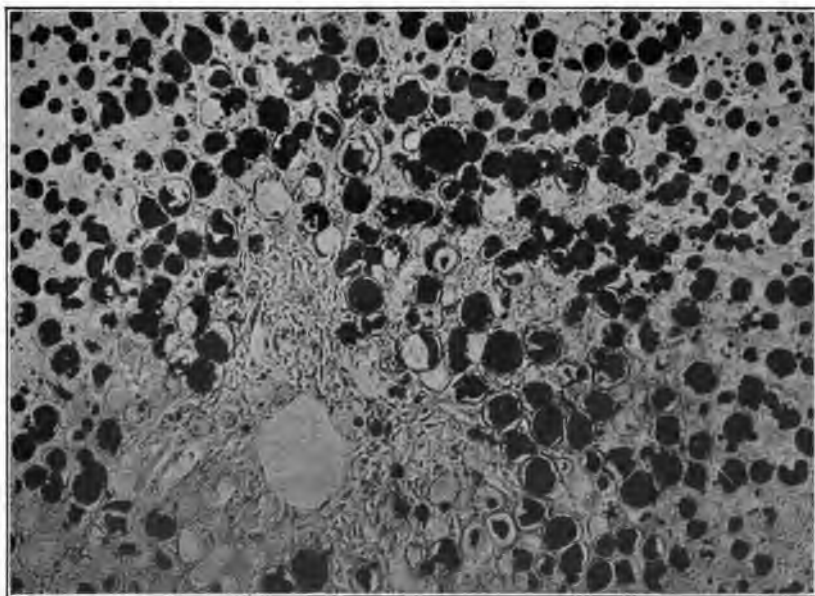


FIG. 2.—MAGNIFICATION 175 DIAMETERS.

LIVER SECTIONS OF ORGANIC-PHOSPHORUS-FED RABBIT NO. 2.

[Fat stained black with osmic acid.]

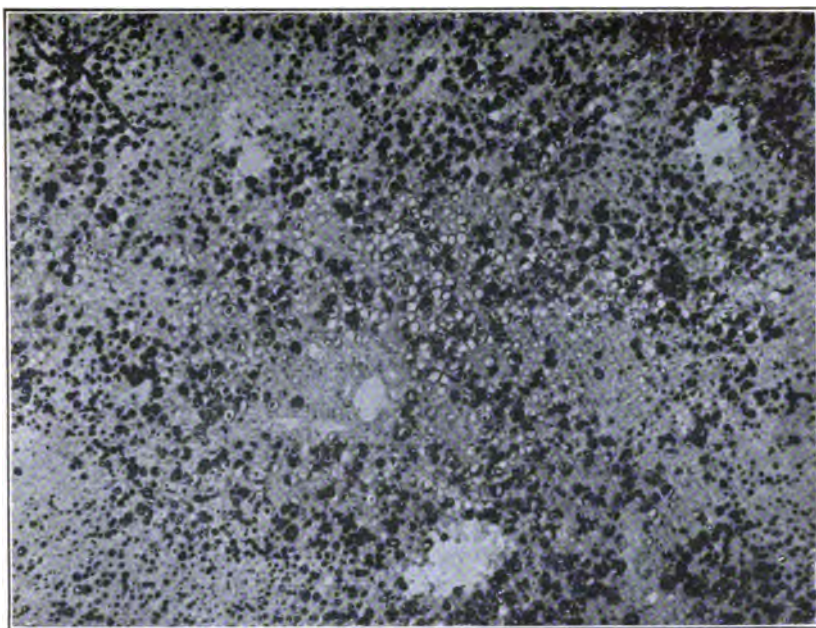


FIG. 1.—MAGNIFICATION 60 DIAMETERS.

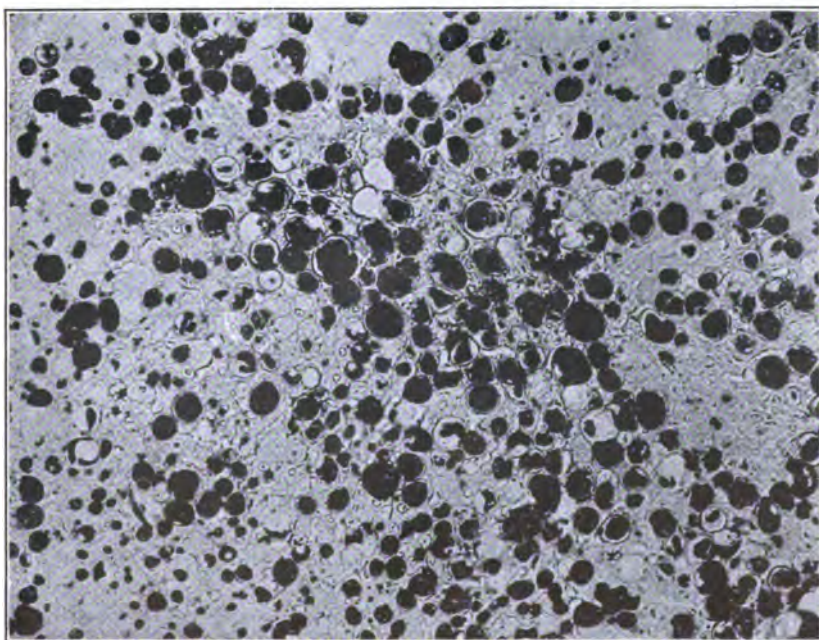


FIG. 2.—MAGNIFICATION 175 DIAMETERS.

LIVER SECTIONS OF INORGANIC-PHOSPHORUS-FED RABBIT NO. 4.

[Fat stained black with osmic acid.]

a smaller proportion of the absorbed nitrogen than did the rabbits fed inorganic phosphorus. With a few exceptions, the daily balances were positive.

The rabbits fed organic phosphorus excreted less phosphorus through the kidneys than did those fed inorganic phosphorus, but they retained a larger proportion of the absorbed phosphorus.

PRINCIPAL FEEDING PERIOD.

The percentages of absorbed nitrogen retained in the case of the rabbits fed inorganic phosphorus show a wide variation, the average figure being practically the same as that for rabbit No. 1 fed organic phosphorus, rabbit No. 2 being excluded from the average. Accordingly, it is impossible to establish any effect produced on the nitrogen metabolism by the two forms of phosphorus fed.

A larger proportion of the ingested phosphorus was eliminated by the kidneys in the cases of the rabbits fed inorganic phosphorus than where the rabbits were fed organic phosphorus. The proportion of absorbed phosphorus retained is greater for the rabbits fed on organic phosphorus. The daily balances were positive with the exception of rabbit No. 2, which died two weeks before the close of the experiment.

The ether-alcohol-soluble phosphorus balances show that more phosphorus in this form was eliminated in the feces where phytin was fed than where inorganic phosphorus was fed.

No ether-alcohol-soluble phosphorus was found in the urine in any case, and it is doubtful if any organic phosphorus is present in the urine of a normal rabbit.

A smaller proportional amount of calcium was eliminated in the urine by the rabbits fed on inorganic phosphorus, but a larger proportional amount was retained in the body than in the cases of the rabbits fed on organic phosphorus. The same statement is true of magnesium.

POST-MORTEM EXAMINATION.

A large proportion of the phosphorus of the fresh intestines of rabbits is dissolved by water during the process of cleaning preparatory to analysis.

The bones of rabbits fed on phosphorus for several months, either organic or inorganic, show a higher content of ether-soluble matter than do the bones of normal rabbits, and they form a larger percentage of the body weight than in the case of normal rabbits.

The livers of the rabbits fed on organic phosphorus for several months show fatty degeneration as well as fatty infiltration. Of the livers of the inorganic phosphorus fed rabbits No. 4 shows both fatty degeneration and fatty infiltration; in the case of No. 3, only slight fatty infiltration is shown. The livers are enlarged and contain

considerably more nitrogen and phosphoric acid than normal livers when calculated to a water and fat-free basis. In these experiments the degeneration and infiltration were most marked when organic phosphorus was ingested.

The brains and nerves of the rabbits fed on organic phosphorus yield a larger percentage of ether-alcohol soluble phosphorus than those of normal rabbits, while the brains and nerves of those fed inorganic phosphorus show lower figures. There is also a larger percentage content of ether-soluble material in the brains and nerves of the phosphorus-fed rabbits than is normal.

The tendency of organic phosphorus, especially in the form of phytin, to produce fat was strikingly indicated in this experiment as in the work of Jordan, Hart, and Patten. The autopsies showed that all the phosphorus-fed rabbits were in an abnormal condition, the livers being especially affected, and the histological examination confirmed the autopsy findings.

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TYPICAL BARLEYS.

1. Two-row barley. 2. Ordinary six-row barley (Manchurian type). 3. True six-row barley.
(Photograph obtained from H. B. Derr, Office of Grain Investigations, Bureau of Plant Industry.)

Issued April 24, 1909.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY —BULLETIN No. 124.
H. W. WILEY, Chief of Bureau.

CHEMICAL STUDIES OF AMERICAN BARLEYS AND MALTS.

BY

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PHYSIOLOGICAL CHEMIST,

AND

ROBERT WAHL,
SPECIAL AGENT.



WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1909.

LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., January 16, 1909.

SIR: I have the honor to submit for your inspection and approval a manuscript containing the preliminary results of investigations specifically authorized by Congress in the agricultural appropriation bill for the fiscal years 1903-1907, to study the barleys grown in different sections of the United States, with a view to improving their quality.

This study is especially valuable at this time, because of the legislation regarding denatured alcohol, for the production of which a certain amount of malt is generally used. The study was made by J. A. Le Clerc, in charge of the vegetable physiological investigations of the Bureau, and Robert Wahl, special agent, with the collaboration of J. S. Chamberlain, T. C. Trescot, A. Given, C. Goodrich, W. J. Young, A. Nilson, N. H. Claussen, and O. Roewade.

I recommend that this report be published as Bulletin No. 124 of the Bureau of Chemistry.

Respectfully,

H. W. WILEY,
Chief of Bureau.

HON. JAMES WILSON,
Secretary of Agriculture.

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STUDIES OF AMERICAN BARLEYS AND MALTS.

INTRODUCTION.

During the past decade many investigations have been undertaken regarding the improvement of the quality of barley for both brewing and feeding purposes. The publication of many of these investigations took the form of discussions as to the relative value of a high-protein and low-protein barley for malting, and thereby additional valuable information has been added to our knowledge of the subject.

Moreover, the still more recent legislation regarding denatured alcohol has given additional impetus to the study of barley and malts. It is well known that for the production of alcohol a certain amount of malt is generally used. This malt is added to convert the starch into sugar, which then can be further converted into alcohol by means of yeast through the ordinary process of fermentation. The amount of malt thus used varies from 5 to 15 per cent of the total amount of raw material employed. The efficiency of the malt depends upon the power of converting starch which it possesses; in other words, a malt is more or less valuable for the production of industrial alcohol according to its diastatic power. When it is remembered that for the production of even 100,000,000 gallons of alcohol (that is, 1 gallon per capita) about 10,000,000 bushels of malt will be required, and, further, that malts vary greatly in diastatic power or the power of converting starch into fermentable sugar, then one easily realizes the full importance of a thorough study of American barleys and malts.

It is generally recognized that the chemist and botanist must work together in order to solve the various agricultural problems, and much work has been done regarding the influence of soil, fertilizers, selection of seed, etc., on the quality of the barley produced; the variety, species, or race of barley to be selected for seed; and the effect of climatic conditions on the properties of the crop. As far as possible these data are presented in such a way as to aid the barley grower and at the same time acquaint the consumer with the properties of barleys grown under different conditions. The results of this work are compared with those of other investigators in order to solve some of the questions which relate to the physical and chemical character-

istics of barley. The investigations herein recorded are purely preliminary. It was thought, however, that the results thus far obtained would be of sufficient interest to warrant their publication without waiting for the completion of the work.

REVIEW OF THE LITERATURE.

The view point from which the various investigations on this subject have been conducted and the conclusions drawn will appear from the following survey of the literature.

Protein and its cleavage products are attracting more and more the attention of those who are investigating barley and malt and their products. On the one hand, preference is given to low-protein barley, rejecting as unsuitable for brewing all barleys containing over 11 per cent of protein. Such barley would be best suited for the production of distillers' malt. Other investigators believe that such a line of demarcation is purely arbitrary and is apt during certain seasons to cause the rejection of barleys which may produce good malts and good beer. As a matter of fact, Prior^a has shown that many Austrian barleys with a protein content of 11 or 12 per cent have furnished superior malts, even for brewing purposes, yielding a high percentage of extract.

The fact that the same variety of barley will vary widely in protein content from year to year, even when grown in the same locality, due to the preponderating influence of environment, would indicate the impracticability of insisting on any such arbitrary standard as the consideration of the protein content alone in accepting or rejecting barley for brewing purposes. Haase, who in 1902 proposed that a good brewing barley should not contain more than 10 per cent of protein, based his conclusions on the fact that Silesian barleys during several years did not average much above 10 per cent. However, in 1905 more than 75 per cent of the barleys examined by Haase contained over 11 per cent of protein. This caused him to adopt 11 per cent as the basis of his system of valuation. This standard refers to the 2-row barley, Hanna and Chevalier, etc., grown in Europe; it does not apply to the ordinary 6-row barleys—the Manchurian or Oderbrucker—grown generally in the Middle Western States, for example, Wisconsin, Minnesota, Iowa, etc., as Wahl has shown.^b The average protein content of the 6-row barleys grown in this country is nearer 12 than 11 per cent, and only rarely is a sample with less than 11.5 per cent of protein produced. Such a standard may easily be accepted in so far as concerns the 2-row barleys or the thick-skin 6-row barleys—that is, the Bay Brewing barleys, grown principally in Cali-

^a *Wochenschr. Brau.*, 1905, 22: 52.

^b Address at the Vienna International Agricultural Congress, 1907.

fornia—and the thin-skin 6-row Utah Winter barley, for all of these varieties contain on an average about 10.5 per cent of protein.

The literature on barley and malt investigations teems with suggestions relating to the kind of barley best adapted for brewing purposes, the influence of various fertilizers on the composition of barley, the changes it undergoes in the process of malting, the rôle which the extract, or the nitrogen, etc., plays in brewing, and the influence of the various constituents on the quality of the finished product. Comparatively little, however, has been written from the standpoint of the production of industrial alcohol.

Attention should be called to the recent fundamental work of H. T. Brown^a and his coworkers on the chemistry of barley and malt in their attempt to establish definite relations between the outward characteristics of barley and the chemical and physiological differences as shown by analysis. They likewise studied the methods of estimating the various nitrogenous constituents of both barley and malt, and the migration of these constituents from the endosperm to the embryo during the process of malting. Their results show that after nine days malting 35 per cent of the nitrogenous constituents of the endosperm become soluble and diffusible and are transported to the embryo. These soluble proteins are present in malt and are found in wort in small amounts. They are supposed by some investigators to exert a relatively large influence on the character of the finished product. Brown has divided those soluble and noncoagulable nitrogenous compounds into six classes: Albumoses, peptones, amidamin, ammonia, organic bases, and residual or undetermined protein. On the other hand, Osborne^b divides the proteins of the whole barley grain, amounting to 10.75 per cent, as follows, with the respective percentage composition given: Leucosin (albumin), 0.3 per cent, equals 2.79 per cent of the total protein; proteose and edestin (globulin), 1.95 per cent, equals 18.14 per cent; hordein, 4 per cent, equals 37.21 per cent; and insoluble protein, 4.5 per cent, equals 41.86 per cent of total protein.

According to Jalowetz^c the basal end of the barley contains more protein than the distal end, the least amount being found in the middle of the berry. In the ear of the plant the right and left longitudinal halves have the same percentage of protein, whereas the grains on the upper half of the heads are richer in nitrogen than those on the lower half, but the amount of nitrogen per individual berry is constant in all sections of the head; the small berries grown on the less perfectly matured heads of the secondary stalks are richer

^a Trans. Guinness Res. Lab., 1903, 1 (1): 96-127.

^b Amer. Chem. J., 1895, 17: 539.

^c Zts. gesam. Brauw., 1906, 29: 172; through Bledermann's Centrbl., 1906, 86: 229.

in nitrogen than are the well-matured grains found on the main stalks, thus showing that a sample of barley even though coming from a single field and representing one variety may vary much in percentage of protein in individual kernels.

Schjerning^a has studied the growing barley plant with special reference to the nitrogenous compounds, analyzing the plants at three different stages of their development, namely, at green, yellow, and full ripeness. Beginning with the formation of the grain, at green ripeness, he analyzed the berries up to the full-ripe stage and concluded that "barley has acquired its full maturity when the conversion of the soluble into insoluble carbohydrates and soluble into insoluble proteins has reached its maximum," and that the ripening of barley is a process tending toward a state of equilibrium in respect to its nitrogenous constituents; when properly ripened barley is harvested very little loss due to respiration takes place during storage. Over ripeness is characterized as a loss of substance. He found that on ripening the percentage of soluble nitrogen in the total nitrogen decreased from 45 to 18 and that the amidamin nitrogen decreased from 28 to 5 per cent. Only traces of proteoses and small amounts of peptones were found. He showed that early harvested barley is poorer in protein than the fully matured grain, and that the chemical composition is not influenced by species, variety, or type of barley, but is affected by the character of the soil and climatic conditions. The length of the growing period, cultivation; and climatic conditions influence the nitrogen content also, and therefore the quality of barley can not be determined from the amount of this constituent alone.

This agrees with the researches of Kukla,^b Jalowetz,^c Prior,^d Wahl,^e and others. These investigators have shown that high protein barleys often give a better malt, which produces a better beer than a malt made from a barley of lower nitrogen content. Kukla also concludes that it is not so much the total protein of barley which influences the quality of the beer as it is the character of the nitrogenous compounds. In his important contribution on the chemistry of barley and malt, Prior^f has shown that the consideration of the amount of hordein (alcohol-soluble protein) and of the insoluble protein constituents of the endosperm is more important than that of the total protein, which should only be considered when above 13 per cent. The hordein he finds located principally near the embryo,

^a Compt. rend. travaux lab., Carlsberg, 1906, 6: 229.

^b Zts. gesam. Brauw., 1900, 23: 418.

^c Ibid., 1906, 29: 172.

^d Wochenschr. Brau., 1905, 22: 52.

^e Amer. Brew. Rev., 1907, 21: 274.

^f Allgem. Zts. Bierbrau. Malzfabr., 1905, 33: 341, 412; through J. Inst. Brew., 1906, 12: 159.

extending to about the middle of the kernel; whereas the insoluble protein is found near the periphery of the endosperm. The best barleys for brewing purposes are those containing a medium amount of protein, namely, from 10.5 to 12 per cent. He finds that the hordein and the insoluble proteins rise in general with the total protein.

The increase of protein is always followed by a decrease of one or more of the other constituents of barley. The opposite is also true. The protein substances, from the standpoint of the brewer and maltster, are now being considered as of the utmost importance, and a relation between them and the starch was one of the first to be noted. Haase showed that an increase of protein was followed by a corresponding decrease of starch. This law was based on results obtained, during several years, from Silesian barleys, and appeared to hold good for this variety. As the relation was not found to be true in regard to other barleys, it has been the subject of much controversy.

It is well known that barleys present differences in physical appearance. Some grains show a mealy or floury endosperm, while the endosperm of others is flinty and translucent. The reasons for these differences and the influence which they exert on malting and brewing and the relation between the character of the grain and the protein content have likewise been the subjects of much study. Brown has observed that steely grains can often be converted into the mealy kind; that is, made mellow, through artificial maturation by steeping or even by weathering after harvest. Johannsen^a long ago showed that the difference between a mealy and a steely or glassy barley was due to the greater number of air spaces in the endosperm of the former, and that in the original condition barleys show no relation between the degree of glassiness and the percentage of nitrogen. In 1868 Jacobsen wrote, in correspondence, that in England it was the general opinion that glassiness and the protein content of barley were related. In 1879 Groenlund wrote an essay in which he showed that early harvested barley can be just as mellow as later harvested barley, and that glassy barley may become mealy by steeping and subsequent drying. He examined 47 different barleys,^b and concluded that glassy barleys did not always contain more protein than mealy ones, but that very often the opposite is true. Schultze^c likewise found no relation between glassy kernels and the nitrogen content, but noted that mealy kernels may contain more nitrogen than steely ones. In 1870 Nowacki^d showed that the difference between a

^a Compt. rend. travaux lab., Carlsberg, 1884, 2: 60.

^b Zts. gesam. Brauw., 1886, 9: 288.

^c Ibid., 1881, 4: 62.

^d Untersuchungen über das Reifen des Getreides, Halle, 1870.

mealy and a glassy wheat was due to the small air spaces imprisoned between the starch granules of the mealy grains and that the specific gravity of the mealy grains was less than that of the flinty. Munro and Beaven^a likewise showed that the specific gravity of mealy kernels is less, due to the larger amount of interstitial air, and that the nitrogen content of such grains is lower, in consequence of which they modify better than do the steely grains. Groenlund^b called attention to the fact that when glassy kernels are steeped and then dried some of the grains become mellow while others remain unchanged. This procedure distinguishes between apparent and real glassiness, and upon this fact Prior^b bases his method for the determination of the degree of dissolution of barley, which consists of the sum of the mealy grains originally present and the percentage of steely grains which become mealy on steeping. This factor shows that the higher the protein content the lower, as a rule, is the degree of dissolution. A later contribution by Prior^c called attention for the first time to the rôle played by the variety of barley on this determination; that what was true of one variety was not necessarily so of another; that is, in some varieties the steely grains are more easily modified than in others. H. T. Brown^b likewise developed a method for the estimation of the coefficient of mealiness, results of which give indication to a certain extent of the value of a barley for brewing or feeding purposes, as he finds that a high coefficient of mealiness is generally accompanied by a low protein content, or vice versa.

Jalowetz^b investigated the relation between the protein content and the character of the endosperm and agreed with other authors that mealy grains are lower in protein than flinty ones. Instead of soaking or steeping the grains for several days at 45° C. and subsequently drying slowly (Brown's method), he suggests that the grain be treated with 40 per cent formalin at the temperature of a boiling water bath for from twenty to thirty minutes. After washing the grains free from formalin and drying them between filters, the character of the endosperm may be immediately examined. This method is claimed by its author to give a good indication of the value of barley. Beaven^d shows that the amount of nitrogen and the quality are closely related, and that high nitrogen barley, accompanied by a steely character of the endosperm, has a higher specific gravity, and that twice as much alcohol-soluble protein is found in such barley as in mealy grains. He also intimates that the nitrogen determination is only useful as an index of quality, other things being equal, and

^a Brown, Trans. Guinness Res. Lab., 1903, 1 (1): 96-127.

^b Loc. cit.

^c Wochenschr. Brau., 1905, 22: 412.

^d J. Fed. Inst. Brew., 1902, 8: 542.

that the size of the grain affects the quantity of extract, the large grains giving more extract than small grains of the same protein content. Beaven considers that the specific gravity of barley may afford a fair index as to quality, and that generally the specific gravity decreases as maturation increases.

Somewhat later Harz^a declared that glassiness of barley is not due to the larger protein content, but to certain kinds of protein substances and to the mechanical combination with the rest of the substances forming the cell. Prior^b separated the different kinds of proteins and determined their relation to one another and their influence on the mellowness of the barley. He found that the causes of the apparent glassiness are the water-soluble nitrogen-free and nitrogen-containing constituents of the endosperm, constituents which are colloidal in character and which cement the starch-containing cells firmly together. When these apparently steely barleys are steeped the cementing constituents dissolve. The real glassiness is due to the cementing of the starch-containing cells by means of the hordein and the insoluble protein. In collaboration with Hermann, Prior^c found that when a 100 per cent steely barley was steeped first in 50 per cent alcohol and then in 75 per cent alcohol at 45° to 50° C., and subsequently dried, the steely barleys became altogether mealy. Previous steeping in water was not necessary. Baker and Hulton^d have recently corroborated Prior's work regarding the fact that permanently or temporarily glassy grains depend upon the presence of nitrogenous or nonnitrogenous colloids.

Until recently most investigators, especially in Europe, have objected to the use of high-protein barley for brewing purposes on the grounds that such barleys give less extract and that this quality is more or less intimately correlated with the flinty character of the endosperm. The latter characteristic is generally held to be an undesirable quality rendering the dissolution of the barley kernels more difficult and resulting in glassy malt. Haase has taken an extreme view of the situation and condemned all barleys containing over 10 per cent of protein. This investigator has, however, gradually receded from his original position, because so many authors have shown that good malts (which produce good beer) could be made from barleys containing much over 10 per cent protein, and furthermore, during 1905, 75 per cent of the Silesian barleys on which he based his argument for low-protein barley contained over 11 per cent of protein.

^a Zts. gesam. Brauw., 1904, 27: 558.

^b Allgem. Zts. Bierbrau. Malzfabr., 1906, 34: 513.

^c Ibid., 1908, 36: 102.

^d J. Inst. Brew., 1907, 13: 328.

Regarding the relation between extract yield and the percentage of nitrogen, Neumann^a substantiates Haase's law that an increase of protein is regularly followed by a decrease in extract. His conclusions are that a good brewing barley should not only contain a low percentage of protein but should give a high extract yield. Furthermore, in high-protein barleys the carbohydrates are more energetically consumed in respiration. On the other hand, a good, distiller's barley may be high in protein, but its most essential quality is its high diastatic power.

Wahl, in his previous writings, has shown that moderately high-protein barleys, when properly malted and brewed, may give even better results than low-protein barleys, as the former are possessed of high vital energy and develop strong enzymatic power during malting, so that the resulting malts are especially rich in both diastase and peptase. The malts from such barleys are not only able to properly saccharify more starch than they themselves contain, but during malting and mashing a comparatively large quantity of protein is rendered soluble by the peptase, the beers produced from such malts being richer in nitrogenous compounds than beers produced from low-protein barley malts.^b

In his work on malt and beer, Evans^c shows that though the nitrogen question is of importance, it is secondary to the study of the starch conversion products produced during malting and mashing. He intimates, however, that much of the color, flavor, and foam of beer is due to the presence of the nitrogenous constituents. Another investigation of importance which should be mentioned is that of Bleisch and Regensburger,^d which showed that the amount of husks increased with the nitrogen, and the loss during malting also grew larger. They advocate the direct determination of the extract as a factor furnishing more reliable data as to the brewing value of barley and malt than does the determination of nitrogen.

Luff's^e work, however, showed no relation between the amount of husks and the percentage of protein. He determined the percentage of husk by treating 150 kernels of barley with 10 cc of 5 per cent ammonium hydroxid in a closed flask, heated in a water bath at 80° C. for one hour. On transferring the kernels from the flask, the husks may easily be separated from the grains. Haase and Bauer^f have shown that a winter barley contains more husks than one with a shorter period of growth and a late ripening variety more than an

^a Wochenschr. Brau., 1905, 22: 98.

^b Wahl, Amer. Brew. Rev., 1904, 18: 485.

^c J. Inst. Brew., 1906, 12: 209.

^d Zts. gesam. Brauw., 1905, 28: 628.

^e Ibid., 1898, 21: 603.

^f Wochenschr. Brau., 1907, 24: 535.

early ripening one. The amount of husks is greater in the starch-poor grains than in full, plump grains, and there is a tendency toward an increase of husks as the weight per 1,000 grains decreases. The husk content does not seem to be influenced by the soil, fertilizers, or width of drills. It is more a varietal characteristic and depends much upon the length of the growing period.

In their paper on conditions affecting the quality of barley Munro and Beaven^a found that the amount of nitrogen in the grain depends more on the character of the season than upon soil conditions, and that the application of phosphates as a fertilizer improves the quality of barley to a greater extent than does the use of potash, soda, or magnesia, while barnyard manure increases the yield, but lowers the general quality. They also found that the lack of color in a barley, which is often due to bad weather conditions, can be remedied by artificial drying. Schneidewind^b showed that with the same conditions of manuring, crops which were high in yield were generally low in protein content. Wein's^c experiments pointed out that nitrogen and phosphates promoted protein formation and that potash influenced the yield and the percentage of starch, thus improving the quality of the crop. He also showed that the barley plant required a large amount of plant food at the early stages of growth.

Voelcher^d showed that the application of nitrogenous fertilizers alone gives a barley of low weight per bushel and of low valuation for brewing purposes, but of high quality from a distiller's standpoint. He quotes Hall as saying that the variety of the barley, rather than the manure used, exerts the chief influence on the nitrogen content. Manuring exerts no influence on the thickness of husks. In this connection Eckenbrecher^e has conclusively shown that climatic conditions and soil exert a far greater influence on the amount of nitrogen, the weight per 1,000, and the weight per bushel than does variety or species. This author grew 6 different varieties of barley in 12 different localities and found that every variety grown in any one locality had very nearly the same percentage of nitrogen, weight per 1,000, and weight per bushel, but that any one variety when grown in the 12 localities showed a marked difference in composition, in size, and in weight of berry; in fact, whereas in one locality a certain type of barley contained 9 per cent of protein, in another locality the protein content was over 14 per cent. Tedin^f also has shown that the protein content is not a race characteristic, and Kiessling^g has demonstrated that the nitrogen content of barley is more dependent on the weather conditions and

^a J. Roy. Agr. Soc., 1900, 11: 185.

^b Wochenschr. Brau., 1905, 22: 20.

^c Zts. gesam. Brauw., 1906, 29: 141.

^d J. Inst. Brew., 1906, 12: 408.

^e Wochenschr. Brau., 1907, 24: 491.

^f Bot. Centrbl., 1907, 104: 383.

^g Zts. gesam. Brauw., 1908, 31: 84.

the nature of the soil than upon variety. The size of grain, however, he considers generally a racial characteristic. In his experiments with barley Reitmair found that phosphorus did not affect the protein content, that there was no relation between the nitrogen of the seed and that of the crop, and that the extract yield and protein content are not transmittable qualities.^a According to Hubert,^b the yield and protein content are dependent on the weather conditions between the flowering and ripening periods, and the application of fertilizers can not overcome the climatic conditions. The same author emphasizes the necessity of having pure barley races, which can be obtained only with the assistance of the botanist. He shows further that pure races will grow more evenly and will give more uniform results on the malting floor.

Regarding experiments on the changes in composition which take place during malting, Windisch and Vogelsang^c showed that in germination and mashing the organic phosphorus of barley and malt is hydrolyzed into the inorganic form. They corroborated the results of Hart and Andrews,^d who showed that there was practically no inorganic phosphorus in barley. Schulze and Castoro^e likewise found that in malting part of the organic phosphorus compounds were converted into the inorganic form soluble in water; that in mashing nearly all of the phosphorus compounds were thus transformed, and that the phosphates found in beer wort were inorganic.

In studying the changes which the proteins undergo during malting and mashing, Weis^f found that the amount of soluble protein increased while the salt-soluble and alcohol-soluble nitrogen compounds, globulin and hordein, respectively, decreased, new compounds with other characteristics being formed in their stead.

Several recent contributions discuss the botanical and physiological characters of the barley plant in more or less detail. Barnstein^g discusses not only the chemistry of barley, its digestibility and use as a food, but also its anatomical characteristics. He shows that the aleuron layer may be two or four cells in thickness, a fact which may have important bearing when high-protein barley is being considered for brewing purposes, for it has been shown that this layer remains practically unchanged during the processes of malting and mashing. Beaven^h brings out the morphological differences between the

^a Vaňha, Kyas, Bukovansky, *Chem. Centrbl.*, 1905, **76**: 695.

^b *Ann. brass. dist.*, 1907, **10**: 347.

^c *Wochenschr. Brau.*, 1906, **23**: 556.

^d *New York Agr. Exp. Sta.*, *Bul.* 238.

^e *Zts. physiol. Chem.*, 1904, **41**: 477.

^f *Zts. gesam. Brauw.*, 1904, **27**: 385, 405, 420, 440.

^g *Landw. Vers.-Stat.*, 1905, **63**: 275.

^h *Loc. cit.*

varieties, giving a classification based on the varying structure. He has shown that the amount of husks or palæ is greater in 6-row barleys and that they act as a protection against mold. Barleys have also been studied from the physical and botanical view points by Lloyd,^a Broili,^b Atterberg and Tedin,^c and in this country by Nilson.^d The last mentioned has shown that the common Oderbrucker or Manchurian barleys are made up of two distinct types, one with short and the other with long haired basal bristles, the former predominating to the extent of about 80 per cent. Besides this difference, it has been noted that the first pair of veins on the husk on the dorsal side of certain barleys are dentated like a saw, while in other grains the veins are smooth. These and other similar morphological differences are used in distinguishing the varieties of barley, but Broili has shown that there are many other varietal differences, for example, the hairiness of the lodicules, all of which must be considered in determining whether a race is pure.

An important study has been made by Wilfarth, Römer, and Wimmer^e on the amount of plant food assimilated by barley during the period of its development in the field. These authors analyzed the growing barley at four different stages of growth for the usual plant constituents and found that at the heading period more potash, soda, and nitrogen are present in the plants per acre than at any subsequent period. The explanation given is that the roots of the growing plant excrete these plant-food elements. More recent investigations by Le Clerc and Breazeale^f showed that the assumption of such an excretion through the roots of growing barley or other plants is erroneous. These authors found that the great loss of plant-food elements noted in barley during the growing period is caused by rain and other atmospheric agencies.

KINDS OF BARLEY.

Barley has been grown for thousands of years. According to Doctor Lauth^g it was grown in China some two thousand years ago, and in Egypt even as far back as six thousand years, as is shown by the pictures of sheaves and ears of *Hordeum herastichum* on ancient coins. It thrives in widely different climates, from Algeria to Nor-

^a Amer. Brew. Rev., 1906, 20: 79.

^b Dissert. Jena, 1906; also J. Landw., 1908, 56: 121.

^c Wochenschr. Brau., 1907, 24: 172.

^d Amer. Brew. Rev., 1904, 18: 413; 1906, 20: 475.

^e Landw. Vers.-Stat., 1905, 63: 1.

^f U. S. Dept. Agr., Yearbook, 1908.

^g Amer. Brew. Rev., 1906, 20: 258.

way and Iceland, and will even grow at an elevation of over 10,000 feet.

The greater portion of the barley grown in this country is 6-rowed, most of which is of the Manchurian type, commonly called "4-rowed barley." This barley is grown principally in the North Central and Middle Western States and the States of the Great Plains. The original source of this barley was Manchuria. From there it was introduced into Germany about 1859, and in 1861 was introduced into Wisconsin, where, on account of its prolific character, it rapidly spread. The barleys discussed in this bulletin may be classified as follows:

The 6-row barleys of the Manchurian and similar types have a relatively high protein content, generally above 11 per cent, the berries being rather small (from 25 to 32 grams per 1,000), with medium thickness of husks. They germinate on the floor in about five days, the malts having rather high enzymic power. Hayduck^a established the fact that a high protein malt has a correspondingly high diastatic power. Such barley is, according to Wahl, especially adapted for the preparation of chill-proof beers and for pasteurized bottled beers. The extract from fine grist may be as high as 75 per cent. The Oderbrucker is similar to the Manchurian in all particulars and was introduced into this country about eight years ago by the Wisconsin Agricultural Experiment Station. Although the malt produced from this barley is quite generally used in brewing in this country, it is especially adapted on account of its high enzymic powers for the production of alcohol.

In the Pacific coast States a similar form, known as "Bay Brewing," is being quite extensively grown. In Utah, and a few local points in other States, there is grown a type of barley locally known as "Utah Winter" (sometimes called "White Club"), with 6 symmetrically arranged rows, which is adapted to brewing purposes. Both of these barleys have a rather low protein content, generally below 10.5 per cent, a high weight per 1,000 (30 to 40 grams), require a longer time for germination, and develop less enzymic power. The fine grist yield of extract from Bay Brewing barley malt is about 68 to 70 per cent, and from Utah Winter, 71 to 74 per cent. The Bay Brewing variety has a thick husk, while the Utah Winter has a relatively thin husk. The 2-row barleys are grown in Montana, Idaho, Colorado, and California. They contain less than 11 per cent of protein on an average, weigh about from 35 to 40 grams per 1,000, have thin husks, require a longer time to germinate than does the 6-row variety of the Manchurian type, and develop less enzymic power. The fine grist yield of extract from malts of Hanna or Chevalier type is from 75 to 80 per cent.

^a Delbrück, J. Inst. Brew., 1906, 12: 643. by Google

Each kind of barley, whether 2-row or 6-row, varies in the number of glassy kernels and in its physical and chemical characteristics according to the conditions under which it grew, the climate playing a prominent part in the production of a low or high protein barley and, in fact, in the production of a first-class barley or one of undesirable quality.

BARLEY VALUATION.

THE BERLIN AND VIENNA SYSTEMS.

To value a barley for malting or brewing is to ascertain the physical, chemical, and physiological properties which it possesses. The maltster, the agricultural distiller, and the brewer are offered all grades of barley. They must know how to judge each, be able to distinguish the favorable or unfavorable factors, and to calculate the value of the product therefrom; and for this purpose various systems of valuation have been evolved. These should not only be exact, but also be simple enough to allow the various factors to be easily and quickly determined. For brewing purposes, malt forms the raw material from which the product is made; from the distiller's view point, malt is but the means to an end. Thus barley is more or less valuable according to the class of malt it will yield and the use to which this malt is put.

The two methods most frequently used for the valuation of brewing barley were the Berlin and the Vienna systems. The former is somewhat older than the latter and depends mainly upon subjective tests—that is, data obtained from outward observation or perception—whereas the Vienna system relies more on objective tests; that is, on data determined in the laboratory by scientific methods. Although these systems are primarily used in valuating barley for brewing purposes, they may be applied to distillers' barley when properly interpreted. Since the systems were first introduced, they have undergone a number of modifications. The Berlin system, as modified in 1908, according to Cluss,^a involves the following factors: (1) Protein in dry substance, (2) color, (3) uniformity, (4) weight of 1,000 grains, (5) fineness of husks, (6) mealiness, and (7) purity of sample. From the sum of these factors are deducted from 1 to 24 points for injured grains, germinated grains, and bad odor.

Each determination is valued on a basis of 9 points, and, in addition, the most important factors, Nos. 1, 3, 4, and 5, are valued on a double basis ($2 \times 1-9$ points). Neumann^b also multiplies the factor "purity" by 2, and Cluss^c gives a double value to "mealiness." It is thus possible for a perfect barley to be rated at 100 points, in

^a Monatsh. Landw., 1908, No. 1.

^b Wochenschr. Brau., 1907, 24: 421.

^c Loc. cit.

which case it is designated as "very fine." When a barley has been given less than 18 points, it is considered bad.

Quality of barley as reckoned by points.

18-30-----	Poor.	67-78-----	Good to fine.
31-42-----	Fair.	79-90-----	Fine.
43-54-----	Medium.	91-100-----	Fine to very fine.
55-66-----	Good.	Over 100-----	Very fine.

Valuation of barley according to protein content.

(Modified Berlin system.)

Protein content.	Designation.	Valuation by points.
<i>Per cent.</i>		
Over 14	Bad.....	1 × 2=2
13.1-14	Poor.....	2 × 2=4
12.1-13	Fair.....	3 × 2=6
11.6-12	Medium.....	4 × 2=8
11.1-11.5	Good.....	5 × 2=10
10.6-11	Good to fine.....	6 × 2=12
10.1-10.5	Fine.....	7 × 2=14
9 -10	Fine to very fine.....	8 × 2=16
Under 9	Very fine.....	9 × 2=18

The protein content together with the weight per 1,000 grains form the two principal factors, and more than any others indicate the extract yield of the malt. Neumann^a considers that they are a better guide in this respect than the determination of extract in barley.

Total number of points obtainable for barley rated according to protein content.

Protein content.	Maximum total valuation.	Protein content.	Maximum total valuation.
<i>Points.</i>	<i>Points.</i>	<i>Points.</i>	<i>Points.</i>
2	16	10	59
4	26	12	70
6	37	14	81
8	48	16	92

The value of having uniform grains is that the barley takes up moisture more evenly on steeping and grows at the same rate on the floor, the dissolution of the endosperm being thus more uniformly effected. The purity of the grain is obtained by shaking 100 grams of barley in a set of sieves, graded at 2.2, 2.5, and 2.8 mm, at the rate of from 210 to 220 revolutions per minute for three minutes. In this way the uniformity factor is also obtained. The greater the proportion of the barley found on any two adjacent sieves the higher is the uniformity factor, or inversely; when less than 50 per cent of the barley is found in sieves Nos. I and II, or Nos. II and III, the

^a Loc. cit.

rating is 1×2 , or 2 points. The more evenly the sample is divided among the three sieves the less uniform it is.

Rating of barley by proportion found on adjacent sieves.

[Modified Berlin system.]

Barley found.	Points as rated.	Barley found.	Points as rated.
<i>Per cent.</i>		<i>Per cent.</i>	
50-60	2×2 or 4	80-85	6×2 or 12
60-70	3×2 or 6	85-90	7×2 or 14
70-75	4×2 or 8	90-95	8×2 or 16
75-80	5×2 or 10	Over 95	9×2 or 18

The weight per 1,000 grains is also doubly valued. The valuation as based on the dry weight of 1,000 grains is shown in the following table:

Valuation of barley as calculated from weight of a thousand grains.

(Modified Berlin system.)

Weight of 1,000 grains.	Points as rated.	Weight of 1,000 grains.	Points as rated.
<i>Grams.</i>		<i>Grams.</i>	
Under 30.	1×2 or 2	43-44.9...	6×2 or 12
30-34.9...	2×2 or 4	45-46.9...	7×2 or 14
35-37.9...	3×2 or 6	47-48.9...	8×2 or 16
38-40.9...	4×2 or 8	Over 49..	9×2 or 18
41-42.9...	5×2 or 10		

The principal change as compared with the former Berlin system is that of giving double value to the protein, uniformity, weight of grain, fineness of husks, and sometimes to purity and mealiness, and making the whole number of points obtainable depend on the protein content. The first Berlin system was restricted to the following tests: Color, weight, uniformity, fineness of husks, mealiness, and purity of samples, together with the negative points, namely, odor, damaged grains, and started grains. In 1897 the protein and weight per hectoliter were added to these subjective tests. Since 1903, under the influence of Haase, the nitrogen factor has become predominant in the Berlin system, this and the size of the grain, or weight per 1,000, constituting the two chief factors of this system.

The present modified and improved Vienna system is based on the following objective factors:^a (1) Weight per hectoliter; (2) weight per 1,000; (3) screenings (assortment); (4) impurity; (5) real steeliness; (6) protein; and on the following subjective factors: (1) Color; (2) uniformity; (3) shape of grain; (4) fineness of husks; (5) general impression, deducting for odor and injured grains.

^a Vorschrift für die Vorbereitung u. Durchführung der Bonitierung der Gerstenprobe, Wien, 1907.

OBJECTIVE FACTORS.

The weight per hectoliter is obtained by actual weighing of the sample. The points given for this factor are as follows:

Valuation of barley according to weight.

Weight of barley.			Rating.
Per hectoliter.	Per bushel.	Per thousand.	
<i>Kilograms.</i>	<i>Pounds.</i>	<i>Grams.</i>	<i>Points.</i>
Over 70	54	Over 38.5	3
67-70	52-54	36.5-38.4	2
66-67	51.3-52	35-36.4	1
Under 66	51.3	Under 35	0

THE ASSORTMENT FACTOR is obtained from the percentage of the sample which passes through a 2.2 mm sieve, and the sample is rated on this point as follows:

Per cent.	Points.	Per cent.	Points.
0-1	4	3.1-4	1
1.1-2	3	4.1-5	0
2.1-3	2		

IMPURITY means the amount of weeds, chaff, dirt, etc., which a sample may contain, and is rated as follows:

Per cent.	Points.	Per cent.	Points.
0-0.2	4	1.1-1.5	1
0.3-0.5	3	Over 1.5	0
0.6-1	2		

REAL STEELINESS or permanent steely grains are given the following points:

Per cent.	Points.	Per cent.	Points.
0-10	6	40-50	2
10-20	5	50-60	1
20-30	4	Over 60	0
30-40	3		

PROTEIN (on dry basis) is rated as follows, all barleys containing more than 14 per cent being rejected:

Per cent.	Points.	Per cent.	Points.
Under 10	6	11.5-11.9	2
10-10.4	5	12-12.9	1
10.5-10.9	4	13 and over deduct	2
11-11.4	3		

SUBJECTIVE FACTORS.

Color is graded as follows: Very good, 3; good, 2; medium, 1; bad, 0.

The uniformity and shape of the kernels is graded: Excellent, 4; very good, 3; good, 2; medium, 1; bad, 0.

The shape for brewing purposes is preferably plump and well closed. Too thin kernels, or even too plump kernels, are of less value.

The fineness of the husks indicated by the wrinkles and folds is given the following values: Especially fine, 6; very fine, 5; fine, 4; less fine, 3; rather coarse, 2; coarse, 1; thick skin, 0.

The factors odor and injured grains are negative; that is, 1 point is deducted for bad odor and 2 points for injured grains. General impression is graded as follows: Excellent, 3; very good, 2; good, 1; bad, 0.

It is seen that the Vienna system relies more on the laboratory and scientific method than does the Berlin system, and is an improvement not only in this respect, but also in that it values the different factors according to their importance and attaches less weight to the protein content of the barley.

According to the Berlin system the principal factors^a in barley valuation are protein content, mealiness, the percentage of husks, and the fineness of husks. Next in importance come the siftings and uniformity of grain, and of least importance are weight per bushel, weight per 1,000, and color. Cluss considers the protein content as the most significant factor, and that the weight per bushel, weight per 1,000 grains, and amount of husks indicate the amount of valuable constituents in barley. He also finds objections to taking the percentage of husks into consideration on the grounds that a properly thrashed barley would contain more husks, and therefore be prejudiced in comparison to short and possibly injured grains.

Haase^b claims that the husks and the shape of the grain afford a certain indication as to quality, but are of secondary importance, stating that, as a rule, the finer the husks the greater the number of damaged kernels.

According to Prior,^c the subjective tests should be considered only in connection with the chemical and physical tests. He believes that the color may indicate the presence of unripe grains or those slightly damaged and browned by bad weather conditions, and the shape may give indication as to variety and fitness for brewing, the plump grains being ordinarily better than the long, thin grains because they contain more starch as well as more nitrogen. The weight per bushel, in connection with the weight per 1,000 grains, is important in showing whether or not a sample consists of light barley and therefore contains less starch and produces less extract. Very heavy grains, however, malt rather stubbornly, and on that account medium-size barley is preferred. Prior would not consider the protein content as of much importance except when above 13 per cent. When below 13

^a Cluss, *Allgem. Zts. Bierbr. Malzfabr.*, 1906, vol. 34, No. 8.

^b *Wochenschr. Brau.*, 1906, 23:35.

^c *Allgem. Zts. Bierbr. Malzfabr.*, 1907, vol. 35, January.

per cent the nature of the protein constituents should be considered. Both Regensburger ^a and Kukla ^b agree with Prior that the quality of the nitrogenous constituents rather than the total nitrogen must be considered in valuing the barley.

Prinz ^c suggests that the points in the valuation of barley should be, first, maturity of the grain, which he considers of greatest importance; second, the protein content; then the uniformity, odor, husks, shape, and damaged grain, in the order named. Uniformity, mellowness, and soundness are more important than color. Furthermore, in all commercial transactions both barley and malt should be bought and sold on the basis of hundredweight rather than per bushel. Hoffmann ^d advocates buying barley and malt on the dry basis, as only dry grain is stable, it being less liable to damage and to attack by mold, besides costing less for transportation. This is certainly a most reasonable proposition, just equally to buyer and seller. It is no unusual occurrence for a grain to lose several per cent of moisture in being transported from one locality to another as, for example, from a humid to a dry climate.

Regarding other criticisms of these European systems, Eckhardt ^e considers the assortment factor obtained by means of the 2.2, 2.5, and 2.8 mm sieves as of the greatest importance, after the degree of mealiness and the amount of protein, as it shows how uniform the grain is. Bleisch ^f suggests that the only criterion in the valuation of barley is a malting experiment on a small scale. Biffen ^g regards a barley of good quality if it is mature, mealy, free from broken and discolored grains, germinates freely and uniformly, and has a good color and a finely wrinkled surface. Heron ^h and Salamon ⁱ consider the diastatic power of malt as an exceedingly useful determination. Besides this, Heron generally estimates the percentage of extract, the specific rotatory power, tintometer value, and moisture, all of which give valuable information concerning malt. Hunicke ^j looks on the physical character of the endosperm as the most important factor, giving greatest weight to the extract content, while Wallerstein considers the loss during malting as the most important determination. As regards the proteins, Wallerstein considers those formed in malting and found in mashing as of greater significance than the total protein. Kreichgauer ^k suggests that the weight per bushel in connection with the biting test will give a good starting point con-

^a Zts. gesam. Brauw., 1905, vol. 28, Nos. 35 and 36.

^b Ibid., 1900, 23: 418.

^c Amer. Brew. Rev., 1907, 21: 589.

^d Wochenschr. Brau., 1906, 23: 534.

^e Zts. gesam. Brauw., 1906, 29: 523.

^f Zts. gesam. Brauw., 1890, 22: 327.

^g J. Inst. Brew., 1906, 12: 345.

^h J. Fred Inst. Brew., 1902, 8: 606.

ⁱ Ibid., p. 2.

^j J. Amer. Chem. Soc., 1904, 26: 1211.

^k Wochenschr. Brau., 1905, 24: 171.

cerning the value of the barley. In a later communication Jalowetz^a recommends that the protein content of the individual grains be taken into consideration instead of the percentage of protein.

A good barley should be sound, have a high germinating power, be rich in starch, and, according to the European system of valuation, low in protein. That the first requisite for good barley is life, high germinating power, and uniform germination needs no discussion, and these may best be obtained by the production of pure races. To both systems there are more or less valid objections made, even by European investigators; though, on the whole, they apply very well to European barleys and conditions. Neither system could, however, be applied in valuing American 6-row barleys, since the conditions both in respect to the type of barley and to the requirements of the brewers are so different in the United States from those prevailing in Europe that the valuation must be made on another basis.

Besides all these factors, a knowledge of the locality of production, the weather conditions prevailing during the growing period and at harvest, the fertilizers used, and the rotation of crops practiced, etc., may aid in estimating the value of barley. For example, it is well known that a late rain discolors the grain and makes it less valuable, and a heavy application of nitrogenous fertilizers tends to increase the protein content, while, on the other hand, much sunshine prevailing during the growing season tends to assure a better grade of barley.

Although all the factors enumerated in both systems are important to a greater or less extent, from a brewer's view point, yet, for the production of alcohol in the agricultural and industrial distillery, some of them may well be given a secondary position. Such factors as fineness of husks, mealiness of endosperm, shape of grain, impurity, and color are of less importance in alcohol production than in the brewing industry, though even these factors are of help in valuing a distiller's barley. Recently harvested barleys have a low germinating power, therefore they should not be malted until at least three months old. The diastatic power of malt is the chief factor when used for alcohol production. This factor is more or less influenced by the characteristics of the grain; namely, uniformity as regards race and age of barley, weight per 1,000 grains, and protein content. A good distiller's barley should have the following characteristics: High germinating power, high protein, uniformity, good color and odor, and cleanness. The malt produced therefrom should possess a high diastatic power, have a pleasant odor, a sweet and agreeable taste, and be free from dirt. As a barley rich in nitrogen is generally one which will yield a malt of high enzymic power—

^a Amer. Brew. Rev., 1907, 21: 590.

in other words, be rich in diastase and peptase—a high nitrogen content of barley is more essential for distillery purposes than for brewing.

ACTION OF THE BERLIN CONGRESS, 1908.

In 1907 the question of barley valuation was considered by the International Agricultural Congress at Vienna, and it was determined to submit it to a special international commission to meet in Berlin in October, 1908. This commission agreed on a general system of barley valuation which, however, was not to be applied to 4 or 6 row barleys. The principles underlying the new international valuation system are:

1. To establish a general system of valuation not considering varieties.
2. To create three grades of value—a highest, a medium, and a lowest.
3. To adopt eleven points for valuation, classified as follows:
 - Highest class:
 1. Protein content (penalties for excessive protein being omitted).
 2. Bad odor.
 - Second class:
 3. Uniformity (as to size).
 4. Weight (1,000 kernels).
 5. Fineness of husk.
 6. Damaged grains.
 - Lowest class:
 7. Color.
 8. Purity of sample (foreign seed).
 9. Sprouters.
 10. Purity as to variety.
 11. Shape of berry.

The following points were omitted from the systems previously described herein:

1. The mellowness of corn, either of the original barley or after steeping.
2. Hectoliter weight.
3. Impression as a whole.
4. Water content of the barley.

The germinating energy was recognized as a valuable point for judging barley, and it was recommended for use at competitive exhibits, but it was considered impracticable for ordinary expositions. This system of barley valuation, as well as the Berlin and Vienna systems which are modified by it, were established for the purpose of serving as guides to jurors of award in judging exhibit barleys, and consequently under circumstances necessitating the judging of large numbers of specimens or samples with dispatch. While in the main the same test points should naturally form the basic features for valuing barley for commercial purposes also, such important points as germinating capacity, the examination for which requires much time, can not well be undertaken for exhibit barleys; besides, exhibits have

usually taken place soon after harvesting, when germinating capacity does not compare favorably with results after proper storage of barley, the higher moisture content alone detrimentally influencing the property of germinating capacity to a decided degree. For this reason and because at the usual exhibit periods moisture content is considerably higher than after storage, it was not included in these systems of valuation. In a commercial system of valuation, however, germinating capacity and moisture content become the main points in the consideration of value, and in the tentative system for American barleys which follows germination capacity forms the basic factor of valuation, to which the importance of all other points or properties is made relative.

TENTATIVE SYSTEM FOR VALUING AMERICAN BARLEY.

The American barleys are to be classified in at least four groups—one comprising the eastern 6-rowed Manchuria barley, cultivated particularly east of the Rocky Mountains; a second, the western 6-rowed barley, the Bay Brewing and Blue barley; a third, the 6-rowed Utah Winter barley; and a fourth, the 2-rowed barleys, the Chevalier, Hanna, Goldthorpe, etc. The western barleys, 2 and 6-rowed, from west of the Rocky Mountains or from the Rocky Mountain territory conform more nearly to the European standard than do the eastern.

For each of these four groups of American barleys a model barley valued at 100 points is used for comparison and more or less points deducted according to the results of each test. A deduction of more than 6 points in any test or division would place a barley below standard.

STANDARD BARLEY.

Standard barley ranges from 94 to 100 points. A barley is below standard when it receives less than 94 points in any one of the examinations of properties described later. For commercial valuation divisions 1 to 8 should be included. For exhibit purposes all tests should be included that are feasible, omitting moisture and germinating capacity for the reasons already given.

The total average of points is found by dividing the sum of the points of each test or division by the number of divisions determined. In this way all divisions need not be included in the test; for instance, moisture, protein, and husk may be omitted by those not having the facilities for making these examinations, and the relative value of the barley nevertheless stands for the remainder of the tests.

CALCULATING THE PERCENTAGES.

The value for each division as stated in points is established by the relative importance of a defection from 100 points, indicating thereby the percentage of inferiority to the assumed model

barley. A barley, for instance, of which 3 per cent do not grow, is rated as 97 for that test or division, a deduction of 1 point being made for each dead berry or germ. The ungerminated barley berries are, however, of greater value than an equal number of grains of wheat or oats, these being too large and heavy to be removed by screening, blowing, or steeping. As wheat or oats may cause protein turbidity in the product, not more than 2 per cent of such grains should be permissible in a standard barley and 3 points should be deducted for every per cent of unremovable foreign matter. For all offal that is removed by screening, blowing, and steeping, only 1 point is deducted for every per cent, because it is not directly harmful. This offal, together with the unremovable foreign matter and the sprouters, should not exceed 6 per cent in a standard barley. This means that a standard barley, after cleaning and skimming, and after deduction has been made for unremovable foreign matter, should yield at least 94 per cent of malting barley.

When valuing barleys from the point of view of the maltster or brewer the deductions for offal should not be included in the final average, which should refer to the cleaned barley. Only for exhibition purposes should the deductions for offal be included in the final average. A barley containing as much as 15 per cent of screenings and skimmings, etc., would only yield 85 per cent of malting barley and could not be considered a standard barley. The 85 per cent of malting barley may, however, be of good or even excellent quality, although probably of low 1,000-berry weight. Its quality is to be determined by the maltster's test (divisions 1 to 12) or the brewer's test (divisions 2 to 14), division 6, offal, being in both cases omitted from the final average. The number of points deducted in one division should be of equal value or importance as indicating inferiority of quality as those in another division. Thus a Manchuria barley with 9 per cent of protein would lose, on account of having 2 per cent less protein than normal, 6 points, and its rate of inferiority would be considered equivalent to that of a barley with 6 per cent of berries not germinated, or with 3 per cent of moisture above normal, or 6 per cent of offal, or 2 per cent of unremovable foreign seeds, or a 1,000-kernel weight of 3 grams below or above the normal. Likewise a barley with 14 per cent of protein, or 2 per cent above normal, would be rated as to inferiority 2×3 points.

This system is equally applicable to all four groups of American barley, but the normal conditions and the requirements to be met by the model barley are somewhat different for each group.

TESTS OR EXAMINATIONS REQUIRED.

For commercial valuation: (a) Merchants' or graders' tests, 1 to 8; (b) maltsters' tests, 1 to 12; brewers' and seed barley tests, 2 to 4.

For exhibit valuation: Tests 2 to 14, excepting 11 and 12.

By subjective examination.

1. Variety and admixtures (Manchuria, Bay Brewing, Utah Winter, Chevallier, etc.) : Deduct 1 to 6 points.
2. Color and brightness : Deduct 1 to 6 points.
3. Odor : Deduct 1 to 6 points.
4. Thickness of husk : Deduct 1 to 6 points.
5. General impression; uniformity of form and size of berries (plump or elongated); thrashing (too close or insufficient); maturity : Deduct 1 to 6 points.

By objective examination.

6. Offal:

By screen: Upper screen: gravel, peas, corn, etc. Lower screen: barley, oats, rye, rape, mustard, etc.

By water: Skimmings, excluding sprouters.

By blowers: Straw, barley, oats, etc.

By cockle machine: Broken kernels, cockle, etc.

(In each case deduct 1 point for every per cent.)

7. Sprouters: Deduct 6 points for every per cent.
8. Remaining foreign matter (wheat, oats, etc.): Deduct 3 points for every per cent.
9. 1,000-berry weight: Deduct 2 points for every gram above or below optimum.
10. Uniformity as to size (the sum of adjacent screens 2.8 mm+2.5 mm, or 2.5 mm+2.2 mm, or 2.2 mm+2.0 mm, giving the highest figure):
 - 100 to 80 per cent deduct 0 point.
 - 80 to 74 per cent deduct 1 point.
 - 74 to 69 per cent deduct 2 points.
 - 69 to 65 per cent deduct 3 points.
 - 65 to 62 per cent deduct 4 points.
 - 62 to 60 per cent deduct 5 points.
 - 60 to 58 per cent deduct 6 points.
11. Germinating capacity: Deduct 1 point for every per cent below 100.
12. Moisture: Deduct 2 points for every per cent above 11 per cent.
13. Protein ($N \times 6.25$): Deduct 2 points for every per cent above or below optimum.
14. Uniformity as to variety (by botanical examination): Deduct 2 points for every per cent of foreign barley or different groups (mixtures of 2, 4, or 6 rowed barleys).
15. Husk (not determined unless considered below standard in subjective examination: Deduct 3 points for every per cent above optimum.

Bushel weight and mealness are not considered. If barley is infested by weevils or other insects or stained or discolored by fungous growths, such as smut, mold, etc., it is absolutely condemned.

PLAN OF THE INVESTIGATION.**SAMPLES AND DETERMINATIONS MADE THEREON.**

The investigation undertaken by this Bureau, of which this is the first report, was authorized by act of Congress, the object being to study barleys grown in different parts of the United States in regard to their use for brewing purposes.

The barleys analyzed comprise 84 samples of the 6-row varieties of Oderbrucker and Manchuria, 18 samples of 2-row varieties, 18 samples of thick-skin, so-called "Bay Brewing" barleys, and 9 samples of the thin-skin Utah Winter. From many of these samples malts, which were likewise subjected to critical analyses, were prepared in malting plants on a commercial scale. Realizing that chemical and physical methods must both be used in the attempt to solve such questions as are involved in the improvement of American barleys, it has been found advisable to make the following determinations on all the barley samples: Water, total nitrogen, soluble nitrogen, coagulable nitrogen, extract, fat, fiber, pentosans, starch, sugars, ash, phosphoric acid, sulphur, lecithins, weight per 1,000 grains, weight per bushel, character of the endosperm before and after steeping, degree of solubility, germinating energy and capacity, amount of husks, bran, endosperm, and embryo. The chemical work, however, is given special prominence in this study, for purely physical analyses alone are not enough to determine the value of barley.

The malt samples were subjected to the following analyses: Water, total nitrogen, soluble nitrogen, coagulable nitrogen, extract (fine and coarse grist), fat, fiber, pentosans, starch, sugars, ash, phosphoric acid, sulphur, lecithins, weight per 1,000 grains, weight per bushel, character of the endosperm, the growth and overgrowth of acrospire, the amount of husk, bran, embryo, and endosperm. It was hoped, from all these determinations, that a better insight as to the changes going on during the process of malting would be gained, and that a guide for future work might be obtained.

CHEMICAL METHODS OF ANALYSIS.

The chemical methods of analysis employed in the Bureau of Chemistry were, unless otherwise described, the official methods adopted by the Association of Official Agricultural Chemists. The exceptions were as follows:

Total sulphur was determined according to the sodium peroxid method.^a

The lecithin determination was made by extracting 10 grams of ground barley or malt with ether, and then extracting the residue repeatedly with absolute alcohol. The ether and alcohol extracts were united, all volatile substances evaporated, and the residue burned with caustic soda to an ash. The ash was then treated in the usual way for phosphoric acid. The amount of phosphoric acid multiplied by 11.37 gives the lecithin content. It is well known that alcohol will extract other phosphorous bodies besides lecithin proper—for example, kephalin; these figures, therefore, include all the lecithin-like bodies soluble in alcohol and ether.

^a Le Clerc and Dubois, J. Amer. Chem. Soc., 1906, 28: 1108.

The soluble proteins were determined by the following method, described by J. S. Chamberlain:

An amount of air-dried barley or malt equivalent to 20 grams of dry material was extracted with water of such a volume that the total resulting mixture amounted to exactly 100 cc. In order to know the volume of liquid in such an extraction it was necessary to determine the volume occupied by the residue from 20 grams of the dry barley after extraction, which was found to be 10.77 cc. In calculating this volume, the figure obtained by H. T. Brown^a for the specific gravity of the dry residue of extracted barley was used, namely, 1.57. Subtracting the volume occupied by the dry residue of extracted barley from the 100 cc gives the volume of liquid actually present. An aliquot of this volume was taken after filtration and the nitrogen determined therein. In practice, however, an amount of barley was taken such that in the proportion of 20 grams of dry substance to 100 cc of the resulting extraction mixture there will be present, after allowing for the volume occupied by the extracted barley, exactly 100 cc of liquid. In this way aliquots of 10 cc, 25 cc, or 50 cc could be easily obtained; that is, 22.41 grams of dry barley in the proportion of 20:100 will require a volume of extraction liquid equaling 112.05 cc, and 22.41 grams of dry barley will leave after extraction a dry residue equaling 12.06 cc. Therefore the volume of liquid present equals $112.05 - 12.06 = 99.99$ cc.

The amount of air-dry barley to be used was then easily calculated in each case from the percentage of moisture in the sample, and this weight of air-dry material was added and extracted under the conditions just described. For the extraction, distilled water at room temperature was used, and the bottles in which the extraction took place were shaken in a revolving shaker for six hours. The mixture was then filtered as rapidly as possible through folded filter papers, the first portion of filtrate, when cloudy, being poured back upon the filter paper until a clear filtrate was obtained. In an aliquot of this clear filtrate the amount of nitrogen was determined, which, multiplied by 6.25, gave the protein, representing the soluble protein.

The soluble noncoagulable protein was determined by boiling 20 cc of the above filtrate over a small flame until the volume was reduced to about 10 cc. After diluting to the original volume, the liquid was filtered, washed, and the noncoagulable nitrogen determined by using the whole of the filtrate.

The soluble coagulable proteins were determined by subtracting the soluble noncoagulable protein from the total protein.

The determinations made by Wahl, which require special mention, were as follows:

For the soluble protein determination 50 grams of finely ground barley were extracted with 250 grams of water for six hours at $18^{\circ} \text{C.} \pm 1^{\circ}$, stirring well every fifteen minutes. The loss on evaporation (approximately 0.1 gram) was made up by adding water until the total weight equaled 300 grams. The extract was filtered clear, maintaining approximately the same temperature. The total soluble nitrogen was determined in an aliquot of the filtrate according to Kjeldahl's method.

The coagulable nitrogen was determined by boiling 100 cc of the above filtrate for thirty minutes, keeping the volume constant, filtering, and estimating the nitrogen in the precipitate. The factor 6.25 was used in all determinations in changing the percentage of nitrogen into protein.

^a Loc. cit.

For the determination of the extract in barley the following method was used by Wahl:

Twenty-five grams of finely ground barley were macerated with 200 cc of distilled water at 65° C. and 25 cc of diastase solution added. The whole was immediately placed in a boiling water bath and kept at that temperature for one hour. The mash was then removed from the bath, boiled briskly for five minutes over a direct flame, stirring continuously, cooled to 60° C., and 75 cc of the diastase solution added, the temperature being kept at 60° to 75° C. for thirty minutes, then raised to 70° C., and held there for another thirty minutes. After inversion, the mash was cooled to from 10° to 15° C., the weight made up to 350 grams with water, and then filtered. The specific gravity of the filtrate was determined by means of the pycnometer. One hundred cubic centimeters of diastase solution were then treated in the same way as was the barley mash, and after cooling were made up to 100 cc, and the specific gravity determined as before. The percentage of extract is calculated as follows:

$$\frac{\left(\frac{W + \frac{MN}{100} + wd}{100} \right)}{B} = c, \quad \frac{(c - ed) 100}{N} = E,$$

in which—

W=weight of water used in the mash.

M=percentage of water in the barley.

N=weight of barley used.

wd=weight of water in the diastase solution used.

B=percentage of extract in mash filtrate according to Balling.

c = extract in 25 grams of barley and 100 cc of diastase solution.

E=percentage of extract in barley.

ed=extract in diastase solution used.

The diastase solution was made by digesting 500 grams of finely ground malt with 2 liters of water for one hour at 15° C.

Wahl's method for the determination of the extract yield^a of the malt was as follows:

Fifty grams of the malt plus 3 kernels are finely ground into the mashing beaker and are macerated with 250 cc of water at 45° C., immediately raised to 45° C., and kept at this temperature for thirty minutes. The temperature is then raised 5° each five minutes until the thermometer shows 70° C. The mash is held at this temperature for thirty minutes. The iodine test is made when the mash reaches 70° C., and is repeated every five minutes until inversion has taken place. The mash is then cooled to about 15° C., and its net weight is made up to 450 grams by the addition of water. The mash is thoroughly mixed, and a quantity of clear wort, sufficient for the saccharometer determination, is filtered through a coarse filter. The liquid is brought to a temperature of 15° C. Its saccharometrical indication is determined by a special Balling instrument standardized at 15° C. and divided into 0.05 per cent. The yield is calculated by the following formula, in which "S" is the saccharometer indication, "H" the percentage of water in the malt (both expressed in percentage of the malt), and "E" the yield of extract:

$$E = \frac{S \times (800 + H)}{100 - S}$$

^a Report of the Analysis Committee, U. S. Brewers' Association, 1902.

The yield of extract on the dry basis E' is computed from " E " by the following formula:

$$E' = \frac{E \times 100}{100 - H}$$

Windisch's extract tables should be employed. The saccharifying or diastatic power of the malt is regarded as very good, if the iodine test shows the absence of starch in the mash, when the temperature has reached 70° C.; as good, if inversion takes place within five minutes, and as fair, if inversion takes place after ten minutes.

To determine the protein dissolved by mashing, 25 cc of mash filtrate were evaporated almost to dryness and nitrogen determined according to Kjeldahl's method, the coagulable protein being determined in the same manner as in barley.

The growth is the ratio of the length of the acrospire to that of the kernel. The determination was made in duplicate by sorting 50 kernels.

The color of the wort was tested by Lovibond's tintometer and the run of the wort by personal judgment. The other determinations were conducted in the same manner as for barley.

MECHANICAL AND BIOLOGICAL METHODS OF ANALYSIS.

The weight per 1,000 grains was determined in the Bureau of Chemistry by means of Kickelhayn's apparatus.

The hulls, bran, embryo, and endosperm were all determined in the Microchemical Laboratory by mechanical dissection of the grain. The method of procedure is described by W. J. Young as follows:

From 6 to 8 good, average grains were selected, and after weighing they were soaked until the hulls could be removed readily, being then subjected to further soaking until the endosperm was completely softened. The grains were finally split lengthwise, and the endosperm removed under water. The hulls, bran, and embryos were placed by themselves in watch glasses, and dried at 100° C. until loss of weight ceased. The water containing the endosperm was allowed to stand in a beaker until the starch settled, when the water was decanted and the sediment dried with gentle heat until moisture was no longer apparent, when the drying was completed at 100° C. More or less loss was observed as a result of this method of drying the endosperm, and this loss was so great in the case of the malts that in these the endosperm was determined by difference.

In the later work on barleys the endosperm was determined by difference in order to obtain results comparable with those obtained from the analysis of the malts.

The physical tests as made by Wahl are as follows: The character of the endosperm was determined by using the Kickelhayn grain cutter. This apparatus cuts 50 berries in two lengthwise at one time. The halves are then easily divided into three groups, namely, those

with a steely endosperm, those that are mealy, and those that are partly steely and partly mealy, or intermediate.

The character of the endosperm after steeping was determined as follows:

Fifty grams of barley were steeped in water at from 15° to 20° C. for twenty-four hours. The water was then poured off and the excess removed from the grains by means of blotting paper. The barley was dried in a drying oven at 30° C. with low draft until the weight approximated slightly less than the original amount taken, about 49 grams. The cutting was done in the same manner as described above.

The germinating energy is represented by the percentage of grains germinated within three days at ordinary temperatures. The germinating capacity is expressed as the percentage of grains which germinated in five days. These tests were made by the ordinary methods for testing germination. The weight per bushel was found by weighing a miniature bushel.

The degree of dissolution was determined by Prior's method:

Steep the barley in distilled water for twenty-four hours at 15° C., drain off the water, removing the excess of moisture by means of filter paper, and dry at 40° C. in an air bath for about two days; then determine the mellowness by means of Kieckelhayn's apparatus. Prior considers the mealy grains which are originally present better than the modified steely grains, and therefore he adds them to the percentage of steely grains modified.

$$A = \frac{(M_1 - M) 100}{100 - M} = M,$$

in which

A = degree of dissolution.

M = per cent of mealy kernels in original barley.

M₁ = per cent of mealy kernels in barley after steeping and drying.

The coefficient of mealiness in steeped and unsteeped barley was calculated according to H. T. Brown's^a formula: Mealy grains are given a value of 100, half mealy 50, and steely 1. The number of grains of each type multiplied by its special value and the sum divided by 100 will give the coefficient of mealiness.

The 1,000 kernel weight is found by counting 500 kernels at random and weighing them on a technical balance. The average of four weighings was taken, unless the difference between the highest and lowest weight of 500 kernels exceeded 0.5 gram, when five or six weighings were taken.

DISCUSSION OF RESULTS.

In discussing the results obtained attention will first be called to the composition of the ordinary 6-row barleys (Table I), the Manchurian and Oderbrucker, calculated to a water-free basis, and then to the change in composition which barleys undergo on being converted into malts. Of the 84 samples of 6-row barleys, the average

^a Loc. cit.

percentage of protein was 11.86, with a variation of from 10.13 to 14.94 per cent; 52 of these samples contained over 11.5 per cent, while only 12 had less than 11 per cent of protein. The sample containing the lowest percentage (10.13) was from Wisconsin, whereas the sample with the highest percentage (14.94) was grown in Montana. The following is a comparative average of the nitrogen results obtained by the Bureau of Chemistry and by Wahl:

Average percentage results on the nitrogen content of three kinds of barley and malt.

Analyst.	Ordinary 6-row.	Western 6-row.	Two-row.
BARLEY.			
Bureau of Chemistry.....	1.91	1.69	1.70
R. Wahl.....	1.93	1.69	1.80
MALTS.			
Bureau of Chemistry.....	1.84	1.56	1.62
R. Wahl.....	1.90	1.59	1.65

PROTEIN CONTENT OF BARLEY.

The following table shows the average amount of protein found in the barleys from several States, beginning with the lowest percentage of protein:

Percentage protein content of barleys arranged by States.

State.	Protein content.	State.	Protein content.	State.	Protein content.
Illinois.....	11.44	Canada.....	11.83	Montana.....	12.63
Michigan.....	11.51	Minnesota.....	11.90	South Dakota.....	12.80
Iowa.....	11.59	Indiana.....	11.94	Kansas.....	13.44
Wisconsin.....	11.64	Colorado.....	12.50	New York.....	14.19
Ohio.....	11.69				

It is thus seen that the North Central States or States of the upper Mississippi Valley produce barleys whose protein content is on an average less than 12 per cent. If it be assumed, as is done in Europe, that a low-nitrogen barley is best for brewing, then these States produce a better quality of barley for this purpose than those grown in Kansas, New York, or South Dakota. On the other hand, the latter States should produce a more nutritious and therefore a better feeding barley and one better suited for the production of denatured alcohol. Clifford Richardson,^a in 1886, found that the Dakota barley was the richest in protein. The average of his results on 60 samples of this cereal is 12.1 per cent, very little higher than the average of 11.86 per cent here reported.

^a U. S. Dept. Agr., Division of Chemistry, Bul. 9.

RELATION OF PROTEIN TO STARCH AND EXTRACT.

It is generally assumed that a high protein grain means a low starch grain, and vice versa. This is true, as a rule, and especially so in the case of wheat. When barleys are considered, however, there are many exceptions, notably the barleys from Ohio, Minnesota, Iowa, and Illinois, which have a comparatively low protein content, and also a rather low starch figure, while those few samples from Kansas and Montana, which contain more than the average amount of protein, likewise show more than the average content of starch. The samples of Indiana, Canada, Michigan, and Wisconsin barleys have a somewhat low protein content, while those from New York, Colorado, and South Dakota have a high protein content. Both of these two groups follow the general expectation, for while the former is high in starch, the latter is low. Thus 33 out of 84 samples of barleys are exceptions to the rule that high protein means low starch, and vice versa. As has been noted, in the case of wheat, protein and starch are generally complementary. With barleys, however, the presence of hulls, varying in amount from 10.2 to 15.4 per cent, makes this point less decisive, though an average of barleys grown under similar conditions shows with a high protein content a lower starch figure. The results indicate that on the whole low-protein 6-row barleys do contain more starch. Fifty-three samples, with an average of 12.2 per cent protein, contained on an average 70.6 per cent of extract, while 31 samples, with 11.1 per cent protein, contained 71.8 per cent of extract. The averages from each individual State do not always show this fact, namely, that there is more extract in low-protein barleys, but if instead of averaging all the samples they be separated into high-protein and low-protein barleys, not taking into account those samples whose protein content is close to the average, then the figures will show that high-protein barleys are low in extract, and vice versa. Twenty-four barleys, with an average protein content of 13 per cent (that is, all barleys over 12.25 per cent), compared with 23 barleys whose average protein content is 10.8 (all samples under 11.25), show 69.94 per cent extract in the former and 72 per cent in the latter. In order, therefore, to bring out the different relations it is often best to take the extreme cases and not regard those which are so near the average that they might be included in one class or the other, according to variations within the limits of error. If, however, only the samples from Michigan, Minnesota, Wisconsin, Iowa, and South Dakota (the States where this type of barley has been found especially well suited to the conditions and where it is therefore extensively grown) be arranged in groups ac-

cording to their protein content,^a a very pronounced tendency in the direction of the theoretical reaction between protein and extract is seen.

Barleys from States of the northern Mississippi Valley showing the relation between protein, extract content, and weight per 1,000 grains.

Number of sam- ples.	Protein.	Extract.	Weight per 1,000 grains.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Grams.</i>
2	10.0-10.5	72.68	28.01
9	10.5-11.0	72.12	27.29
19	11.0-11.5	71.63	26.97
13	11.5-12.0	71.15	26.39
9	12.0-12.5	70.70	26.07
9	12.5-13.0	70.19	25.66
4	13.0-13.5	70.16	26.14
4	13.5-14.0	70.71	26.14

Only the last group, containing from 13.5 to 14 per cent of protein, forms an exception to the general rule that the percentage of extract decreases with an increasing protein content. The table further indicates that there is a greater decrease in extract for barley containing from 10 to 12 per cent of protein than in that containing from 12 to 14 per cent.

From these results it is very evident that high-protein barleys of the 6-row type give low extract yields, a fact which has been observed by many others, especially in regard to 2-row barleys.

RELATION OF PROTEIN CONTENT TO WEIGHT PER 1,000 GRAINS.

Neumann^b showed that low-protein 2-row barleys were generally of higher weight and that they produced more extract. The results here shown indicate also that low-protein barleys of the 6-row type weigh somewhat, though very little, more per 1,000 grains, thus again corroborating Neumann's work. This is shown by the last column of the preceding table, which gives the average weight of 1,000 grains within the different groups. While it would appear from the table as if the weight of 1,000 grains varies more or less irregularly, especially for barleys containing from 12 to 14 per cent of protein, there is an obvious tendency for the weight of 1,000 grains to decrease as the protein content of the barley increases from 10 to 12 per cent, though there are many individual exceptions.

If one considers those samples of approximately the same percentage of protein, it will be found that invariably the heavier contain the

^a Three samples have been left out which either contained more than 14 per cent of protein or had abnormally large berries, together with much protein, owing to special cultivation and breeding.

^b Wochenschr. Brau., 1905, 22:98.

most extract, a fact already established by Neumann^a and Kunz.^b This is well illustrated in the following table:

Relation between weight and extract content for barleys of the same protein content.

Protein.	Number of samples.	Weight per 1,000 grains.	Extract.	Protein.	Number of samples.	Weight per 1,000 grains.	Extract.
<i>Per cent.</i>		<i>Grams.</i>	<i>Per cent.</i>	<i>Per cent.</i>		<i>Grams.</i>	<i>Per cent.</i>
10-10.8.....	2	28.0	72.6	12.0-12.4.....	5	27.5	71.8
	2	27.5	72.4		4	25.2	70.2
10.8-11.....	4	27.8	72.5	12.4-12.8.....	4	26.9	70.9
	3	26.7	71.5		4	25.0	69.5
11-11.2.....	5	28.0	72.4	12.8-13.2.....	4	27.8	71.0
	4	26.1	71.1		4	25.9	69.8
11.2-11.6.....	4	27.7	71.9	13.2-13.8.....	4	26.8	70.9
	3	24.5	69.8		3	26.3	69.1
11.6-11.8.....	5	27.8	71.8	Over 13.9.....	3	28.2	70.3
	4	25.0	70.6		4	26.4	66.0
11.8-12.0.....	4	27.3	72.1				
	4	25.5	70.6				

The figures show that a high-protein barley may give a high extract, provided the weight per 1,000 grains is large, and vice versa. The size of the grain affects, therefore, the quantity of extract, other factors being equal. This table also gives the relation between the protein and extract content, showing the natural tendency for high-protein barley to give less extract. There are, however, many individual exceptions to this rule, as was found by Prior^c in his work on 2-row barley.

RELATION OF THE PROTEIN CONTENT TO THE CHARACTER OF THE ENDOSPERM.

Not only do the low-protein barleys weigh more per 1,000 grains and contain more extract, but they are much more mealy after steeping. For example, 31 samples of barley with an average protein content of 11.1 per cent have a coefficient of mealiness of 84, while 53 samples whose average protein content is 12.2 per cent have a coefficient of mealiness of only 80. This difference is accentuated if only those samples which contain over 12.2 per cent of protein are compared to those containing less than 11.25 per cent. In this case the former have a coefficient of mealiness of only 77 as compared with 87 for the latter.

Yet the actual number of flinty grains in the samples, before steeping, is about the same in each class, the high-protein samples containing 16 mealy and 43 steely grains per 100 and the low-protein barleys containing 15 per cent mealy and 44 per cent steely. The behavior

^aAddress at the meeting of Versuchs- und Lehranstalt für Brauerei, October, 1906.

^bWochenschr. Brau., 1906, 23: 530.

^cLoc. cit.

of these two classes of barley on steeping is, however, quite different. The steely and half-steely grains of the low-protein barleys are changed during this process to a greater degree than are those of high protein content. This fact is more definitely brought out by comparing the samples of high and low protein content after steeping. For example, 24 samples with more than 12.25 per cent of protein gave a coefficient of mealiness of 73.8, while 15 samples with less than 11 per cent of protein gave a mealiness coefficient of 87.8.

The same samples before steeping contained 17 per cent steely and 40 per cent mealy, with a coefficient of mealiness of 61.7, and 14 per cent steely, and 42 per cent mealy, with 64.1 coefficient of mealiness, respectively. These results show that permanent steely grains are richer in protein, and if not carefully malted they furnish steely malt and sinkers.^a The high-protein barley underwent a 24 per cent modification on steeping, while the low protein barleys were modified to the extent of 35 per cent. The estimation of the coefficient of mealiness is important only when made after steeping. As the high-protein barleys contain a larger percentage of steely grains and have a lower coefficient of mealiness than the low-protein barleys, it follows that steely grains contain less extract than do mealy ones. This is shown in the following table:

Ordinary 6-row barleys compared as to the character of the endosperm.

LESS THAN 75 PER CENT OF MEALY GRAINS AFTER STEEPING.

State.	Number of samples.	Extract.	Weight per 1,000 grains.	Degree of dissolution.	Coefficient of mealiness.	Protein.	Fat.	Starch.
		Per cent.	Grams.			Per cent.	Per cent.	Per cent.
Canada.....	3	71.7	26.9	68.5	74.5	12.0	1.95	59.2
South Dakota.....	4	69.2	24.0	71.5	75.5	13.3	1.72	58.2
Indiana.....	1	71.9	24.3	69.2	80.1	12.1	2.00	62.0
Iowa.....	2	70.1	24.9	51.3	64.7	12.6	2.05	57.1
Kansas.....	1	69.6	27.8	48.4	66.1	12.5	1.85	62.2
Michigan.....	2	71.6	26.5	61.0	75.0	11.4	1.97	59.1
Minnesota.....	11	70.7	26.0	72.7	78.2	12.2	2.02	58.9
Montana.....	3	68.8	23.8	67.9	76.6	12.6	2.20	60.1
New York.....	1	70.9	27.9	64.9	73.1	14.3	1.99	56.4
Wisconsin.....	19	71.3	27.2	74.3	78.0	12.0	2.03	58.8
Average.....	(47)	70.7	26.6	71.0	76.5	12.2	2.00	58.8

MORE THAN 75 PER CENT OF MEALY GRAINS AFTER STEEPING.

Colorado.....	1	72.1	28.8	80.3	83.0	11.7	2.07	56.8
South Dakota.....	1	69.5	24.1	95.2	92.0	12.4	1.76	62.4
Illinois.....	1	72.0	28.2	82.1	89.0	12.0	2.05	56.6
Iowa.....	5	70.7	24.7	103.6	91.3	11.4	2.00	59.0
Michigan.....	4	71.1	25.9	83.8	88.9	11.6	2.03	60.6
Minnesota.....	9	69.2	26.7	89.1	89.0	12.1	2.03	57.8
Wisconsin.....	15	72.1	28.7	95.2	89.0	11.4	1.98	59.8
Ohio.....	1	71.1	26.2	89.1	88.0	11.9	1.92	57.0
Average.....	(37)	71.0	27.1	92.4	86.5	11.6	1.99	59.2

^a J. Brand, Zts. gesam. Brauw., 1906, 29: 661.

The same relation of protein content to permanent and transitory steeliness is plainly shown by the following table, in which all samples examined are arranged in groups according to their protein content:

Effect of steeping on mealiness considered from the view point of protein content.

Number of samples.	Protein.	Degree of dissolution (Prior).	Coefficient of mealiness (Brown).		
			Before steeping.	After steeping.	Difference.
	<i>Per cent.</i>				
2	7.0-7.5	107.7	31.00	98.75	67.75
1	8.0-8.5	100.9	11.80	99.50	87.70
5	9.0-9.5	108.4	36.65	96.31	59.66
6	9.5-10.0	92.8	11.66	94.26	82.60
6	10.0-10.5	90.7	22.47	90.69	68.22
13	10.5-11.0	87.3	31.61	87.39	55.78
20	11.0-11.5	82.4	33.55	83.51	49.96
20	11.5-12.0	84.3	34.26	84.44	50.18
13	12.0-12.5	73.6	34.35	78.82	44.47
13	12.5-13.0	66.7	31.15	75.12	43.97
6	13.0-13.5	60.6	32.09	70.71	38.62
7	13.5-14.0	67.9	33.23	74.47	41.24
1	14.0-14.5	64.9	29.59	73.19	43.60
3	14.5-15.0	62.9	47.12	71.14	24.02
1	15.0-16.0	54.3	44.85	69.58	24.73

The last column, which gives the difference between the coefficients of mealiness before and after steeping, indicates that as a general rule the more kernels are transformed into the mealy state by steeping, the less protein the barley contains. Both the degree of dissolution (Prior) and the coefficient of mealiness after steeping (Brown) increase with decreasing protein content; that is, the lower the protein content of a barley the more mealy in general its structure. If the figures for degree of dissolution be compared with those indicating the coefficient of mealiness (after steeping) it is seen that they are nearly identical for such barleys as are ordinarily used for malting purposes—that is, those containing from 10 to 13 per cent of protein—whereas beyond these limits the degree of dissolution rises or falls more rapidly than the coefficient of mealiness.

RELATION OF PROTEIN AND HULL CONTENT.

Prior^a has also indicated that no connection exists between the protein and the hull content of barley. This may be true of 2-row barley, but when the 84 samples of 6-row barleys are examined one easily sees that with an increase in protein content there is also a corresponding increase in the percentage of hulls. Twenty-four samples of high-protein barleys (average, 13 per cent of protein) contain 12.9 per cent of hulls and 11.8 per cent of bran, while 23 samples of low-protein barleys (average, 10.9 per cent of protein) contain 12.4 per cent of hulls and 11.6 per cent of bran.

^a Through Pure Products, 1907, 3: 92.

This may be due to the fact that the smaller grains contain relatively more protein than the larger ones, and of course the small grains contain relatively more hulls. Beaven^a showed that small berries gave less extract, because they had a relatively larger amount of hulls.

COMPARATIVE COMPOSITION OF LARGE AND SMALL GRAINS.

One expects to find a rather close relation between the size of the grain and the amount of starch present. The difference, however, is really very slight, especially when the extreme cases are compared. For example, 20 samples the weight of which per 1,000 grains was over 28.5 grams have 58.6 per cent of starch, while 31 samples under 27 grams per 1,000 grains contain 58.8 per cent of starch. Johannsen's^b results, showing that the big grains contain relatively less nitrogen than the small grains of the same variety, is also corroborated.

The percentage of embryo and bran in small and large grains varies little, but the results seem to show that small grains contain a lower percentage of endosperm than do the larger ones. Twenty samples with an average weight of 30.3 grams per 1,000 grains contain 72.5 per cent of endosperm, whereas 31 samples of smaller grains (average weight per 1,000 grains, 25.6 grams) contain 71 per cent.

There is also a relation between the size of the grain and the amount of hulls, the larger grains containing somewhat less hulls. This has also been found to be true by Wallerstein^c and Beaven.^d

Large grains contain more extract, starch, and endosperm than do the small ones. In 20 samples of 6-row barley, with a 1,000-grain weight above 28.5 grams, the percentage of bran is 11.8; hulls, 12.2; embryo, 2.5; endosperm, 72.5; extract, 71.7; starch, 58.6; and the weight per bushel is 49.5 pounds; while 31 samples of smaller grains of the same variety—that is, those weighing less than 27 grams per 1,000 grains—contain about 11.6 per cent of bran, 13 per cent of hulls, 2.53 per cent of embryo, 71 per cent of endosperm, 70.7 per cent of extract, 58.9 per cent of starch, and have a bushel weight of 46.6 pounds. The larger grains contain also less fiber, pentosans, and ash, but have a higher coefficient of mealiness. There is no appreciable difference in the fat, sulphur, or lecithin content in large and small grains. The weight per 1,000 grains varied from 19.9 to 33.5 grams. The weight per bushel varied from 42.5 to 51.5 pounds. The light grains are less plump, contain more nitrogen, and produce less extract. On the other hand, extra heavy grains are

^a J. Fed. Inst. Brew., 1902, 8: 542.

^b Compt. rend. travaux Carlsberg, 1899, 4: 122.

^c Communications from Laboratory and Scientific Station for Brewing, Sec. Ann. Rep., 1904.

^d Loc. cit.

richer in extract material, but, according to Prior, they malt less easily. The weight per bushel is not so important as is this weight taken in connection with the weight per 1,000 grains.

As has already been stated, the weight per bushel varies from 42.5 to 51.5 pounds, with an average of 46.7, the sample of lowest weight per bushel being from Montana and having also the lowest weight per 1,000 grains and a high percentage of nitrogen. This relation of high protein content to low bushel weight has been observed by many investigators, and almost invariably occurs when the sample has, for some reason, failed to develop normally and fully. It is a well-known physiological fact that the protein of cereals develops to a very large extent comparatively early in the life of the plant, whereas assimilation and the formation of carbohydrates may proceed as long as the leaves or stem contain any green coloring matter. If for any cause the plant fails to develop a plump grain it will naturally show not a larger amount of nitrogen but a relatively higher percentage. The barleys from Ohio and Illinois, which contain the lowest percentage of nitrogen, are characterized by being the heaviest. The weight per 1,000 grains likewise shows a very wide variation, 19.9 grams to 33.5 grams, with an average of 26.9 grams, the smaller grain showing a somewhat higher percentage of nitrogen.

OTHER CONSTITUENTS OF BARLEY.

The percentage of pentosans shows a variation from 8.31 per cent in Ohio-grown barley to 11.51 in the sample from South Dakota. These results indicate that a high content of hulls is accompanied by a high percentage of fiber and of pentosans, as would be expected, since grains with a high content of fiber generally yield the most pentosans, because of a rather close connection, not necessarily genetic, between fiber and pentosans.^a High-protein barleys contain the most pentosans, on an average.

The percentage of fiber varies from 4.34 to 6.68, with an average of 5.76 for all samples. The average found by Clifford Richardson was only 4.08. These variations from one year to another are probably due to weather conditions. It may be interesting also to note that the sample grown in New York contained the least amount of hulls (only 10.17 per cent), while the one from Montana contained the largest amount (15.36 per cent). The high-protein barleys are somewhat richer in fiber than are the low-protein barleys, thus corroborating the researches of Bleisch and Regensburger.^b

The percentage of fat in the barleys grown in the different States varied from 1.67 to 2.46, with an average of 2.02 per cent for all

^a Calabresi, *Staz. sperim. agrar. ital.*, 1906, **39**: 69.

^b *Zts. gesam. Brauw.*, 1905, **28**: 628.

samples. Richardson ^a states that the average of 10 samples was 2.87 per cent, considerably higher than that found in any of the present samples.

König ^b showed that the fat content of barley varied from 1.35 per cent, obtained in barleys grown in Württemberg, to 2.97, the latter representing the average of 16 Russian-grown barleys. There is no appreciable difference in fat content between high and low protein barleys. This corroborates Neumann's ^c results on 2-row barley.

The sugar results obtained in this investigation, though interesting, are unsatisfactory, owing to the fact that it was impossible to determine the sugar in the barleys until the samples were considerably over a year old. The barleys were harvested in the fall of 1904, and sent to the Bureau of Chemistry in the summer of 1905, soon after which most of the other determinations were made. In the fall and winter of 1905 the sugar determinations were begun. The results obtained were normal; that is, the invert sugar content varied from 0.8 per cent to 2.03 per cent, while the cane sugar varied from 1.02 per cent to 5.09 per cent. In February, 1906, while these results were being obtained, work had to be suspended temporarily and was not resumed until the following May, during which interim it was found that all of the invert sugar and most of the cane sugar had disappeared. This was true of both the ground and the unground barley. The cause of this phenomenon remains unknown, though it may be closely connected with the loss of diastase which takes place when seed has lost its germinating power. ^d

The percentage of ash in these 84 samples of barley averages 2.98 and varies from 2.5 to 3.5 per cent, a rather large variation, the Tennessee, Ohio, Illinois, and New York barleys containing less than those from the other States. On the other hand, Montana and Kansas barleys are very high in ash. No relation exists between the ash content and the percentage of protein. Delbrück ^e found that high and low protein barleys gave practically the same percentage of ash, fat, and hulls. The average ash content found in 79 samples of American barley, as quoted by König, is 3.10 per cent, a figure quite close to that obtained on the samples reported here.

The percentage of phosphoric acid varies from 0.8 to 1.25, increasing and decreasing as a rule with the amount of ash, in which the percentage of phosphoric acid varies from 27.4 to 42.1, the largest amount being found in Ohio. When extreme cases are taken into consideration there seems to be also a rather close relation between the amounts of phosphoric acid, protein, and starch present; the higher the per-

^a U. S. Dept. Agr., Division of Chemistry, Bul. 9.

^b Untersuchung landwirtschaftlich und gewerblich wichtiger Stoffe, p. 486.

^c J. Inst. Brew., 1907, 18: 87.

^d Albo, through J. Inst. Brew., 1908, 14: 405.

^e Through Trans. Amer. Brew. Inst., 1905, 8: 16.

centage of phosphoric acid the less protein and the more starch. This was true in over two-thirds of the samples examined. In this connection Richardson^a found that phosphoric acid fertilizers increased the number of mealy grains, the effect being just the opposite to that of nitrogen fertilizers, which increase the flinty characteristics. It may thus be quite possible to decrease the protein content of barley by the liberal use of phosphate fertilizers; in other words, since it is fairly definitely known that nitrate fertilizers increase the protein content of grains, phosphates may be used to increase the starch content, thus producing a low protein barley. Kunz,^b however, could find no relation between the amount of phosphoric acid and the extract yield.

The amount of sulphur varies in the 84 samples of barley from 0.15 per cent to 0.256 per cent, with an average for all of the samples of 0.182 per cent, following the protein content closely. As sulphur is a natural constituent of protein, it might be expected that a high-protein barley would contain more sulphur than one with low protein, and that this is the case was shown in over 80 per cent of the samples.

RELATION OF TOTAL TO SOLUBLE PROTEIN.

Regarding the soluble protein, the results indicate that the greater the total content of protein the smaller the percentage which is soluble; in other words, a larger proportion of the total protein is soluble in low-protein barleys than in high-protein barleys. The following table will show how general this relation is when the barleys are divided into groups according to their protein content:

Relation between the total and the soluble protein content of barley.

State.	Number of samples.	Protein content (below 11.49).		Number of samples.	Protein content (11.50-11.99).	
		Total.	Proportion soluble.		Total.	Proportion soluble.
		Per cent.	Per cent.		Per cent.	Per cent.
Canada.....				3	11.8	16.6
South Dakota.....				1	11.9	19.0
Iowa.....	4	11.1	19.2	1	11.6	19.6
Illinois.....	1	11.4	18.4			
Indiana.....				1	11.9	18.0
Michigan.....	3	11.1	18.0	2	11.8	17.3
Minnesota.....	5	10.9	17.6	4	11.9	18.5
Montana.....	1	11.4	19.2	1	11.6	17.0
Ohio.....				1	11.7	19.4
Wisconsin.....	18	11.0	17.6	5	11.6	17.7
Average.....	32	11.0	17.9	9	11.8	17.9

^a U. S. Dept. Agr., Division of Chemistry, Bul. 9.

^b Wochenschr. Brau., 1906, 28: 530.

Relation between the total and the soluble protein content of barley—Cont'd.

State.	Number of samples.	Protein content (12-12.49).		Number of samples.	Protein content (over 12.50).	
		Total.	Proportion soluble.		Total.	Proportion soluble.
		Per cent.	Per cent.		Per cent.	Per cent.
New York.....				1	14.2	17.1
Colorado.....				1	12.5	15.7
South Dakota.....	2	12.3	17.8	2	13.7	16.3
Iowa.....	1	12.1	18.7	1	13.1	18.0
Michigan.....	1	12.2	17.3			
Kansas.....				1	13.4	13.1
Minnesota.....	8	12.3	18.0	3	12.9	18.5
Montana.....				1	14.9	16.8
Wisconsin.....	5	12.2	16.7	6	13.3	17.5
Average.....	17	12.2	17.0	16	13.4	17.1

In general, the 2-row barleys and the western barleys also show that a somewhat larger proportion of the protein is soluble in low than in high protein barleys (see p. 49). The same relation is true with respect to the malts also.

In the following table the samples of the Manchurian-Oderbrucker type are arranged in groups according to protein content and the averages of soluble and of soluble-coagulable protein given for each group. There is a small but distinct decrease of the percentage of soluble protein with increasing total protein, but there are many individual exceptions to this rule, especially in the case of the maximum and minimum figures obtained.

Relation between the soluble, the soluble-coagulable, and the total protein of some Manchurian-Oderbrucker barleys.

Total protein in barley.	Soluble protein (in terms of total protein).			Soluble-coagulable protein (in terms of soluble protein).
	Average.	Maximum.	Minimum.	
	Per cent.	Per cent.	Per cent.	Per cent.
10-11	16.8	17.8	15.4	27.9
11-12	16.7	19.1	14.6	29.7
12-13	16.5	19.6	14.0	28.7
13-14	16.0	18.6	14.8	29.1
14-15	15.4	15.7	15.1	31.7

As has already been noted, the amount of soluble nitrogen decreases with the increase of the total nitrogen; Wallerstein showed, however, that high protein and high soluble protein go together. This is not necessarily a contradiction, for in this study the percentage of soluble nitrogen was compared with the total nitrogen, while Wallerstein has only compared the percentage of total soluble nitrogen of high and low protein barleys.

Of the proteins in barley, therefore, from 81 to 85 per cent are insoluble. Of the soluble protein, about 30 per cent are coagulable. On

the other hand, Evans^a finds that with 2-row barleys about 50 per cent of the soluble protein is coagulable, which is a much larger amount than that obtained in the work here reported on ordinary 6-row barleys.

LECITHIN IN ITS RELATION TO PROTEIN AND PHOSPHORIC ACID.

The amount of lecithin, or rather of alcohol-and-ether soluble bodies, varies from 0.39 to 0.69 per cent, with an average of 0.53 per cent, in accordance with the protein content. Stoklasa^b found that seed containing the most protein likewise held a higher percentage of lecithin. This is substantiated by these data with but few exceptions; the amounts are, however, too small to make it possible to draw many conclusions.

There is no apparent connection between the amount of phosphoric acid in barley and the lecithin content, probably due to the fact that barley, like wheat, contains a larger proportion of a water-soluble organic phosphorus compound, similar to, if not, phytin, and that the amount of phosphorus found in barley is more nearly related to this more abundantly occurring body than to lecithin, the latter phosphorus compound being present in only relatively small quantities.

From the results given in Tables I and II it is seen that fully 35 per cent of the ash is composed of phosphoric acid compounds of which less than 5 per cent is in the form of lecithin phosphoric acid. The bulk of the phosphorus is present in the barley as a calcium-magnesium-potassium salt of oxymethylene diphosphoric acid, as was shown by Hart and Andrews,^c who also showed that practically no inorganic phosphorus compounds existed in grains. The latter statement was afterwards corroborated by Schulze and Castoro,^d and more recently Windisch and Vogelsang^e established the same fact in regard to barley. There are several organic compounds of phosphorus existing in plants, chief among which, besides the previously mentioned compound, phytin, are the lecithin-like bodies, which have a glycerin radicle, and the nucleins, which are protein compounds containing phosphorus. Phytin occurs in quite large amounts, while the two latter compounds are present in smaller quantities.

Calcium, magnesium, and potassium, the more important ash constituents besides phosphorus, form on an average about 2.7 per cent, 7.3 per cent, and 23 per cent, respectively, of the total ash. There appears to be no appreciable difference in the amount of these constituents in the ash of low-protein barleys and high-protein barleys.

^a J. Inst. Brew., 1906, 12: 209.

^b Ber. deut. chem. Ges., 1896, 29: 2761.

^c New York Agr. Exp. Sta., Bul. 238.

^d Zts. physiol. Chem., 1904, 41: 477.

^e Wochenschr. Brau., 1906, 23: 516.

COEFFICIENT OF MEALINESS.

There is also a very noticeable difference in the degree of dissolution and in the coefficient of mealiness in these samples of barley. The former varies from 37 to over 117, while the latter shows a variation of from 55 to 98. Prior's method of determining the degree of dissolution very often gives values over 100. The coefficient of mealiness as determined by Brown gives somewhat lower results on low-protein barleys and higher results on high-protein barleys than Prior's degree of dissolution. The two methods are fairly indicative of the quality of barley. If the samples be arranged according to the percentage of mealy grains found after steeping, separating them into two classes, those with more than 75 per cent of mealy grains and those with less, it is seen (p. 39) that the lower protein content and the higher number of mealy grains go together. There is no difference in the fat content, but there is somewhat more starch in those samples containing the high percentage of mealy grains. The weight per 1,000 grains and the amount of extract are also greater in high than in low percentage mealy grains.

SUMMARY OF RESULTS.

ORDINARY SIX-ROW BARLEYS.

High-protein 6-row barleys contain a larger percentage of fiber, pentosans, hulls, bran, and embryo, but a smaller percentage of starch, extract, and soluble protein. The percentage of soluble protein that is coagulable is somewhat greater in high-protein than in low-protein barleys, but in the case of 2-row and Bay Brewing barleys there is no appreciable difference in this respect. The high-protein barleys weigh less per 1,000 grains and per bushel, besides having a lower degree of dissolution and coefficient of mealiness. This applies to all varieties of barley analyzed. No appreciable difference can be noted between high and low protein barleys in their content of fat, ash, sulphur, and lecithin, as is shown in the following tables, which also show no difference in the percentage of steely grains before steeping between barleys of more than and less than 11.5 per cent protein, when these are averaged. However, if the extreme cases—that is, those samples containing more than 12.25 per cent protein—are compared with barleys of less than 11 per cent protein, we find that the former contain 10 per cent steely and 63 per cent mealy grains after steeping, while the latter have only 4 per cent steely and 77 per cent mealy, thus clearly showing that the barleys with over 12 per cent protein are not so easily altered by the process of steeping as are low-protein barleys.

Comparison of ordinary 6-row barleys of high and low protein content.

MORE THAN 11.5 PER CENT PROTEIN.

State.	Number of sam- ples.	Fat.	Fiber.	Pentosans.	Starch.	Ash.	Sulphur.	Phosphoric acid.		Calcium oxid.		Magnesium oxid.		Potassium oxid.		Lecithin.	Hulls.		Bran.		Embryo.	Endosperm.		Weight per 1,000 grains.	Extract.	Weight per hecto- liter.	Degree of dissolu- tion.	Coefficient of meal- iness.	Before steeping.		In total protein.																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																												
								P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.		P.p.d.	P.p.d.	P.p.d.	P.p.d.		P.p.d.	P.p.d.						P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.	P.p.d.

LESS THAN 11.5 PER CENT PROTEIN.

Iowa.....	3	1.89	5.91	10.5	59.1	3.0	0.17	1.00	0.08	0.23	0.69	0.47	12.8	11.8	2.7	72.7	24.2	70.0	60.3	100.9	91.4	21	38	16.5	24.7	11.2
Michigan.....	5	2.02	5.58	9.5	59.0	2.9	0.18	1.03	0.10	0.22	0.69	0.57	12.0	11.5	2.7	71.9	26.3	71.2	60.2	74.3	83.2	15	46	17.0	28.6	11.3
Minnesota.....	6	2.05	5.80	9.7	58.9	2.9	0.17	1.07	0.08	0.20	0.69	0.50	12.7	12.0	2.5	72.3	26.4	71.3	61.4	80.8	77.7	13	42	17.2	27.7	11.1
Montana.....	1	2.10	5.40	8.8	62.3	3.3	0.20	1.10	0.09	0.24	0.73	0.65	11.2	11.2	2.7	71.8	28.4	70.6	62.3	66.7	77.1	8	58	16.8	28.1	11.4
Wisconsin.....	16	2.00	5.60	9.4	59.9	3.0	0.18	1.07	0.08	0.22	0.70	0.51	12.3	11.5	2.5	72.6	27.9	72.6	61.6	87.0	85.4	17	38	16.7	29.2	10.9
Average..... (31)		2.00	5.66	9.56	59.6	2.96	0.18	1.06	0.08	0.22	0.70	0.52	12.3	11.6	2.5	72.4	27.0	71.8	61.2	84.4	83.8	15	44	16.9	28.4	11.1

The following table likewise shows the difference in coefficient of mealiness, degree of dissolution, percentage of extract, etc., between high and low protein barleys:

Comparison of high and low protein barleys.

TWO-ROW.

State.	Number of samples.	Degree of dissolution.	Coefficient of mealiness.	Protein.	Weight per thousand.	Weight per bushel.	Ex-tract.	Soluble protein.	Coagu-lable protein in water-soluble
OVER 11.5 PER CENT OF PROTEIN.				Per cent.	Grams.	Pounds.	Per cent.	Per cent.	Per cent.
California.....	1	62.0	77.6	12.9	32.6	52.7	74.0	14.3	32.4
Colorado.....	5	48.7	54.3	13.2	41.6	51.0	71.4	15.8	32.8
New York.....	1	77.8	83.5	12.6	31.3	50.2	72.7	16.5	31.8
South Dakota.....	1	50.0	68.1	11.6	28.3	48.5	69.4	17.7	21.8
Kansas.....	1	77.1	79.1	17.4	34.7	47.2	69.4	15.4	34.9
Average.....	9	56.7	64.4	13.4	37.2	50.4	71.4	16.0	31.6
UNDER 11.5 PER CENT OF PROTEIN.									
California.....	1	116.9	97.5	9.3	38.0	49.5	73.9	15.8	28.8
Germany.....	1	100.8	98.0	9.5	47.6	55.7	79.1	15.2	30.3
Canada.....	1	77.2	87.0	10.8	38.1	50.5	74.2	18.4	31.3
Idaho.....	1	85.0	91.0	11.0	37.5	52.5	73.7	15.4	31.8
Montana.....	5	116.4	97.6	10.0	38.9	55.0	76.4	19.0	32.3
Average.....	9	95.7	95.7	10.1	39.5	53.7	75.9	17.6	31.5

BAY BREWING BARLEY (6-ROW).

OVER 11.5 PER CENT OF PROTEIN.									
California.....	2	39.4	61.0	13.2	32.0	40.3	68.3	13.5	25.4
Oklahoma.....	1	54.3	69.6	15.8	28.0	39.2	62.9	20.3	33.7
Kansas.....	1	79.6	81.1	13.6	29.7	40.0	68.4	14.9	25.8
Tennessee.....	2	55.4	66.2	12.2	28.5	45.7	71.4	15.5	31.3
Average.....	6	53.9	67.5	13.4	29.8	41.9	68.5	15.5	28.8
UNDER 11.5 PER CENT OF PROTEIN.									
California.....	6	100.7	96.2	9.1	36.4	46.5	71.8	15.4	27.7
Idaho.....	3	96.2	96.8	10.1	40.0	46.7	72.2	16.3	29.9
Washington.....	3	97.6	97.5	10.0	40.8	47.2	72.3	16.2	29.4
Average.....	12	98.8	96.6	9.5	38.4	46.7	72.0	15.8	28.7

The great variations in the composition of these 84 samples of 6-row barleys, which belong practically to the same variety—that is, Manchurian—and which have been grown in widely separated localities differing from one another in soil and general climatic conditions, again demonstrate how great is the influence of environment on the composition of plants. There is a greater difference in the composition and in the physical characteristics of the barleys of the same type grown in different localities than there is between different varieties grown in the same environment. This is well illustrated in Tables I and II, which give the average composition of the four varieties of barley. There seems to be a greater influence exerted by climate than by seed, variety, or difference in soil characteristics. Ac-

cording to König,^a a barley grown in a sandy soil, a clay soil, and a soil rich in lime differed in protein content as follows: 11.1, 13.4, 12.7. Much larger differences than these are obtained by growing the same varieties in different localities. Eckenbrecher^b likewise has recently shown that there is a greater difference in composition and physical characteristics of barley of the same type grown in different localities than of different varieties grown in the same locality. The same has been shown by Kiessling^c and others.

TWO-ROW BARLEYS.

No attempt will be made to draw conclusions of this character from the data here presented on 2-row, Utah Winter, or Bay Brewing barleys, because of the fact that comparatively few samples of each variety were analyzed. In general, however (Table II), it is readily seen that the percentage of protein is very slightly lower in the 2-row barleys than in the 6-row barleys (Table I) of the Manchurian type, the average in the former case being about 11.6 per cent. The five samples from Montana average less than 10 per cent. Although the 2-row barleys do not contain much less protein than do the 6-row, there appears to be somewhat less fiber, pentosans, ash, sulphur, hulls, embryo, and steely grains, but more starch, extract, soluble albumen, bran, and endosperm, a higher coefficient of mealiness and degree of dissolution, and a greater weight per 1,000 grains and per bushel in the 2-row than in the 6-row barleys. The other constituents show no great variation between the two types of barley. A striking difference between 2-row and 6-row barleys is found in the fact that the former contain a larger proportion of bran than of hulls, while in the latter the percentage of hulls is greater than the percentage of bran. The western 6-row barleys are in this respect similar to the ordinary 6-row barleys.

SIX-ROW WESTERN BARLEYS.

Twenty-seven samples of 6-row western barleys were analyzed. They are usually called Bay Brewing barley or Utah Winter, the former being characterized by their thick skin and the latter by their somewhat thinner hulls. Both varieties are large. Compared with the ordinary 6-row barleys, they show a closer resemblance to them in chemical composition than do the 2-row barleys. They are, however, much larger, weigh more per bushel, and contain less protein (somewhat less than the 2-row barleys). They contain a slightly larger percentage of hulls, but less total sulphur and soluble protein than do other 6-row barleys. The average weight per 1,000 grains of 6-row barley is less than 27 grams, compared with 36 grams

^a Untersuchung landwirtschaftlich gewerblich wichtiger Stoffe, p. 517.

^b Wochenschr. Brau., 1907, 24: 491.

^c Loc. cit.

as the weight per 1,000 grains of western barleys. There is a very close agreement between the two varieties in the content of ash, fat, fiber, bran, pentosans, starch, and ash constituents.

COMPARISON OF MALTS.

Thirty malts were prepared from the ordinary 6-row barleys, 8 from the western barleys (Bay Brewing and Utah Winter), and 5 from 2-row barleys.

In comparing the composition of malts made from these three different varieties of barley, Table III shows that 6-row malts contain a larger percentage of the following constituents: Sulphur, lecithin, total protein, soluble protein, soluble noncoagulable protein, and embryo; a smaller percentage of starch, of extract in fine and coarse grist, and of bran; a smaller weight per bushel and per 1,000 grains, and a smaller coefficient of mealiness. Two-row malts, on the other hand, are higher in endosperm, weight per bushel, extract in fine and coarse grist, and coefficient of mealiness, and lower in fiber, pentosans, hulls, and embryo. The large western malts—the Bay Brewing and Utah Winter—are higher in hulls and lower in sulphur, protein, soluble protein, and soluble noncoagulable and coagulable protein. There is very little difference between the varieties in the percentage of ash, phosphoric acid, and fat. The western malts resemble those of the ordinary 6-row barleys in the amount of fiber, pentosans, hulls, bran, embryo, endosperm, and starch which they contain. They are somewhat like the 2-row malts in weight per bushel, weight per 1,000 grains, and in the per cent of total protein.

The following table shows how high and low protein malts compare in weight per 1,000 grains, per cent of extract in fine grist, weight per hectoliter, per cent soluble protein and per cent coagulable protein, the per cent mealiness, and coefficient of mealiness. On an average, the high-protein malts are much less mealy in the case of all three classes of malt. The coefficient of mealiness is also less, as are also the weight per 1,000 grains, the per cent of extract, and the per cent of soluble protein.

Comparison of high and low protein malts.

State.	Number of samples.	Protein.	Mealy.	Coefficient of mealiness.	Weight per 1,000 grains.	Extract of fine grist.	Weight per hectoliter.	Soluble protein in total protein.	Coagulable protein.
OVER 11.5 PER CENT OF PROTEIN (6-ROW).									
		<i>Per ct.</i>	<i>Per ct.</i>		<i>Grams.</i>	<i>Per ct.</i>	<i>Kilos.</i>	<i>Per ct.</i>	<i>Per ct.</i>
South Dakota.....	1	13.3	71	80.0	21.0	71.7	46.7	38.0	3.7
Illinois.....	1	12.1	95	97.0	24.9	72.6	45.4	36.8	4.8
Iowa.....	1	13.0	76	79.7	22.7	72.1	45.8	37.6	3.2
Michigan.....	1	12.6	76	85.1	21.4	71.5	48.0	33.4	3.5
Minnesota.....	5	12.3	76	84.4	24.5	73.5	48.2	36.7	4.0
Ohio.....	1	11.5	78	84.6	23.3	72.9	47.1	41.3	3.4
Wisconsin.....	7	12.5	80	90.4	24.7	73.4	44.3	35.3	3.4
Colorado.....	1	11.6	60	76.1	28.3	73.2	51.3	29.4	5.9
Average.....	18	12.5	80	86.6	24.0	73.1	47.0	36.4	3.8

Comparison of high and low protein malts—Continued.

State.	Number of samples.	Protein.	Mealy.	Coefficient of mealliness.	Weight per 1,000 grains.	Extract of fine grist.	Weight per hectoliter.	Soluble protein in total protein.	Coagulable protein.
UNDER 11.5 PER CENT OF PROTEIN (6-ROW).									
		<i>Per cent.</i>	<i>Per cent.</i>		<i>Grams.</i>	<i>Per cent.</i>	<i>Kilos.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Michigan.....	2	11.2	91	95.0	25.9	75.7	48.7	37.0	4.0
Minnesota.....	4	11.1	78	86.0	23.7	72.0	47.0	37.6	5.2
Wisconsin.....	7	11.3	94	96.4	25.0	73.9	46.7	37.8	4.6
Average.....	13	11.2	89	93.0	24.7	73.6	47.1	37.6	4.7
OVER 11.5 PER CENT OF PROTEIN (2-ROW).									
New York.....	1	13.8	60	73.6	22.7	72.9	48.7	35.6	4.5
UNDER 11.5 PER CENT OF PROTEIN (2-ROW).									
Montana.....	4	9.6	96	97.7	33.5	78.7	53.1	38.0	4.3
OVER 11.5 PER CENT OF PROTEIN (BAY BREWING AND UTAH WINTER).									
Washington.....	1	12.3	68	81.6	35.1	71.8	50.0	32.2	3.6
UNDER 11.5 PER CENT OF PROTEIN (BAY BREWING AND UTAH WINTER).									
California.....	2	8.2	90	93.0	34.0	72.0	45.4	38.3	3.6
Idaho.....	2	10.1	91	95.0	32.9	72.6	46.3	32.3	3.3
Utah.....	1	10.9	65	77.6	32.8	73.9	48.7	32.3	4.0
Washington.....	1	8.3	93	97.5	34.5	77.2	48.7	35.6	3.5
Montana.....	1	9.5	86	91.5	32.3	75.3	45.4	36.3	3.9
Average.....	7	9.3	87	91.8	33.4	73.6	46.6	35.1	3.5

The hulls of 6-row malt form a much larger percentage of the grain than do the hulls of 2-row malts, and yet the protein content of the 6-row barley is 1.5 per cent higher than that of the 2-row variety. These two factors only emphasize how much smaller the percentage of carbohydrates must be in 6-row than in 2-row barley malts.

Malts are sometimes rejected by brewers because of a high bushel weight. The following table will show that this factor is absolutely useless when considered alone, for very often those malts having a high bushel weight will give a larger yield of extract in the coarse grist than malts of lower weight per bushel.

Comparison of weight per bushel and yield of extract.

Kind of malt.	Number of samples.	High weight per bushel.	Extract.	Number of samples.	Low weight per bushel.	Extract.
		<i>Pounds.</i>	<i>Per cent.</i>		<i>Pounds.</i>	<i>Per cent.</i>
2-row malt.....	4	41.25	76.7	2	36.5	70.6
6-row Bay Brewing malt.....	4	38.25	70.1	4	35.6	69.9
6-row Manchurian malt.....	15	37.7	70.5	12	35.6	69.9

As Wallerstein^a has shown, a high bushel weight of malt is no more an indication of inferiority than is the low weight per bushel a

^a Communications from Laboratory and Scientific Station for Brewing, Sec. Ann. Rep., 1904.

proof of its superiority. This clearly shows that one factor alone, and especially the bushel weight, is not enough to determine the value of a malt. Again, many times, even when this factor is considered in connection with the weight per 1,000 grains, there are not sufficient data at hand to warrant a rejection of the malt, for the following table will illustrate how it is possible to have malts whose bushel weights are high, but whose weights per 1,000 grains are low, and yet the extract yield is higher than the average. On the other hand, some malts with a high weight per 1,000 grains and a high bushel weight give a yield of extract lower than the average. The average weight per 1,000 grains of malts of high bushel weight and of high yield of extract is very little higher than in the case of low bushel weight.

Comparison of 6-row malts having a high and a low weight per bushel.

High weight per bushel.			Low weight per bushel.		
Weight per bushel.	Weight per 1,000 grains.	Extract.	Weight per bushel.	Weight per 1,000 grains.	Extract.
<i>Pounds.</i>	<i>Grams.</i>	<i>Per cent.</i>	<i>Pounds.</i>	<i>Grams.</i>	<i>Per cent.</i>
38.75	25.9	74.1	36.25	21.0	68.8
36.75	26.0	70.8	35.25	24.9	71.5
37.25	21.4	68.5	35.50	22.7	67.5
36.50	24.7	63.6	34.25	22.1	69.8
38.50	24.8	71.6	36.00	25.3	68.2
40.50	24.6	70.0	35.75	24.6	70.7
37.50	23.0	69.3	36.00	24.3	71.8
37.50	23.3	71.0	35.75	25.1	73.0
38.00	25.0	70.8	36.00	25.0	69.1
37.00	25.1	71.9	35.25	25.5	70.6
37.00	25.0	72.5	36.25	24.0	66.0
37.75	24.8	70.0	34.75	22.7	71.9
36.50	25.4	68.3			
38.00	27.3	70.9			
37.50	23.4	70.8			
37.7	24.6	70.5	35.6	23.9	69.9

As a general rule, however, a malt with a high weight per bushel will give more extract and will weigh more per 1,000 grains than a malt with low weight per bushel. The following figures selected from the preceding table plainly show this:

A comparison of the extremes of weight per bushel with yield of extract.

Over 37.7 pounds per bushel.		Less than 35.6 pounds per bushel.	
Weight per 1,000 grains.	Extract.	Weight per 1,000 grains.	Extract.
<i>Grams.</i>	<i>Per cent.</i>	<i>Grams.</i>	<i>Per cent.</i>
25.9	74.1	24.9	71.5
24.8	71.6	22.7	67.5
24.6	70.0	22.1	69.8
25.0	70.8	25.5	70.6
24.8	70.0	22.7	71.9
27.3	70.9		
25.4	71.2	23.5	70.2

But it is not always enough to know the weight per bushel and the weight per 1,000 grains in order to properly select malt. One should also determine other factors, such as mellowness, percentage of germination, water, protein, and extract, the color, odor, impurity, and the diastatic power, etc., basing the decision on all of these results.

From the entire study it is very evident that the variation in climatic conditions throughout the country, the difference in soil, the different methods of cultivation and rotation practiced—all have their bearing on the characteristics of barley, and from the great variation in composition it is safe to assume that the United States can produce barley of the first rank, whether 6-row or 2-row varieties be grown. As the climate varies greatly from one locality to another, and such conditions exert the greatest influence on the quality of the crop, care should be taken to select the seed and locality according to the type of barley desired. For example, moist climates and localities where plants have long periods of growth, especially between the stage of flowering and maturity, generally produce a low-protein barley. In such localities it would generally be impossible to grow barleys rich in protein.

CHANGES IN COMPOSITION DURING MALTING.

One of the most interesting and instructive series of results obtained in this work relates to the changes which each constituent of the barleys underwent during malting. This is shown in Table IV. These figures were obtained by analyzing the malts, and then calculating the malt analyses to the basis of the corresponding barleys by multiplying the results of the malt analyses by the factor obtained by dividing the weight of 1,000 grains of malt by that of 1,000 grains of barley. This factor averages about 89, but as a rule the factor found by actually weighing the barley and the amount of malt obtained therefrom on a laboratory scale was used. In several cases, however, the factor 89 was used in the conversion of the malt figures to the basis of the barley. This was the case wherever the results showed that an apparent error had been made, or where a sample of either the malt or barley had been lost before the weight per 1,000 grains was obtained. The loss in malting a barley is due to the loss of soluble constituents and respiration of carbonic acid and to the formation of the radicles. On the other hand, there is a slight gain in weight due to the fixation of water during the conversion of starch to sugar, and possibly also to the hydrolysis of the proteins.^a The losses on malting were then calculated by dividing the difference between the percentage of each constituent in the barley and in the malt (calculated to the barley basis) by the percentage of that constituent in the barley itself.

^a Long, J. Amer. Chem. Soc., 1907, 29: 295.

In this way the following changes due to malting were estimated, the figures given being the average of the results obtained from the analyses of 43 samples of barley and of their corresponding malts:

Loss and gain in the various constituents of barley due to malting.

Constituent.	Gain or loss.	Constituent.	Gain or loss.
	<i>Per cent.</i>		<i>Per cent.</i>
Fat.....	- 7.7	Sulphur.....	+ 9.0
Fiber.....	- 8.4	Lecithins.....	+ 34.3
Pentosans.....	- 1.6	Hulls.....	- 8.5
Starch.....	- 28.0	Bran.....	- 37.0
Reducing sugars as invert sugar.....	+ 400.0	Embryo.....	+ 78.7
Cane sugar.....	+ 71.0	Endosperm.....	- 10.2
Ash.....	- 20.7	Total protein.....	- 12.0
Potassium oxid.....	- 48.0	Soluble protein.....	+ 72.5
Calcium oxid.....	- 22.0	Soluble-noncoagulable protein.....	+ 104.0
Magnesium oxid.....	- 17.0	Soluble-coagulable protein.....	+ 13.0
Total phosphoric acid.....	- 12.7		

Although it should not be assumed that these results represent exact amounts, yet they indicate fairly well the changes going on during malting. From these averages it is seen that when barleys are malted they lose appreciably in fat, lime, magnesia, phosphoric acid, hulls, protein, fiber, and endosperm. There is a greater loss, however, in starch, ash, bran, and potash. No appreciable loss is noted in pentosans, while on the other hand there is a considerable gain in the amount of lecithin and soluble coagulable protein, and a very large increase in sugars, embryo, soluble protein, and soluble noncoagulable protein.

The loss of the different constituents is, of course, due to the growth of the acrospire and rootlets or malt sprouts, to the amount of respiratory products produced during this growth, and to the various physiological changes; for example, the conversion of starch into maltose, etc. Thus, much of the starch (over 20 per cent) has been lost during malting, but most of this loss is made up by the corresponding gain in sugars. A part of the sugars produced from this starch and some of the fat were given off as carbon dioxid produced by respiration during the malting. Another part of the starch conversion products was transferred to the malt sprouts, the insoluble and therefore the immovable starch having first been converted by the diastase into soluble and transferable sugars, which then migrated to the sprouts. These sprouts are likewise rich in phosphoric acid and other salts containing over 1.5 per cent of phosphoric acid alone, besides over 3 per cent of potash and an appreciable amount of lime and magnesia. This partly accounts for the large loss of ash, phosphoric acid, and other constituents, and also of the bran of barley during malting. As one can readily see, the loss of phosphoric acid, of ash, and of bran are closely related, being, respectively, 12.7 per cent, 20.7 per cent, and 37 per cent. The ash not only lost phosphoric acid but also some of all of the other constituents, namely,

48 per cent of potash, 22 per cent of lime, and 17 per cent of magnesia. This explains why the loss of ash is greater than that of phosphoric acid. According to König,^a barley bran contains about 7 per cent of ash, of which 50 per cent is phosphoric acid. That fact explains why such a loss in bran takes place during the process of malting barley. A large portion of the ash lost, consisting of phosphoric acid and other salts, would naturally come from the bran, which constituent of barley is richest both in ash and in phosphoric acid. The protein lost during malting is, of course, to be found chiefly in the malt sprouts. In order to be thus transported from the barley grain to the malt sprout, the insoluble protein had to be made soluble by the proteolytic enzymes which are always present in grains and only await propitious conditions in order to become active. The insoluble protein, having been converted into soluble and movable protein, and possibly also having been changed into the amid form, migrates to the growing plantlet and rootlet and again becomes insoluble and fixed, just as the starch first becomes soluble before it can migrate, and through physiological processes again becomes insoluble in forming cellulose for the cell walls, etc. It should be noted that high-protein barleys lost on an average over 16 per cent of the total nitrogen, while the low-protein barleys lost about 11 per cent on malting, the former losing considerably more starch also during this process.

Analysis of the sprouts or rootlets obtained from malted barley showed that they contained about 5 per cent of the total phosphoric acid, 20 per cent of the potash, and from 2 to 3 per cent of the lime and magnesia found in the malt. The difference between the total loss observed in malting and the above figures shows the amount of these constituents actually lost on steeping.

Brown and Morris^b show that in malting there is an increase of over 300 per cent of cane sugar in the embryo and a still larger increase in the endosperm, besides which a large amount of maltose is found in the malt endosperm, having been produced from the starch of the barley endosperm. Similar results were obtained by O'Sullivan^c long before in his masterly researches on sugars. Delbrück^d quotes Grüss and Schönfeld's work showing that despite the fact that enzymes are breaking down the starch into sugar during malting, a considerable reconversion of starch from the sugar takes place, especially during the drying of the malt. Hoffmann and others (see Delbrück) showed that the drying of malt likewise changed amids into protein. This has led to a comparison of the physiological process taking place

^a Untersuchung landwirtschaftlich und gewerblich wichtiger Stoffe, p. 771.

^b Text-book of Science of Brewing, p. 74.

^c J. Chem. Soc., 1886, 49: 58.

^d J. Inst. Brew., 1906, 12: 644.

during the drying of the malt with that occurring during the ripening of grains. Some work carried on in the Bureau of Chemistry on the changes in sugar and soluble nitrogenous constituents during malting and during the drying of the malt have failed to fully corroborate the above conclusions relating to the conversion of sugar into starch. The results show, on the dry basis, the following amounts of sugar (calculated as dextrose): Barley soaked two days preparatory to malting, 1.16 per cent; green malt, 6.14 and 7.25 per cent; and malt (dried one day at 35° C.), 8.3 and 11.47 per cent. An increase rather than a decrease in sugar has evidently taken place during the first day's drying, due probably to the fact that the slow drying at 35° C. with the large initial amount of moisture in the green malt was really a continuation of the malting process which continued until the moisture content became too low, due to evaporation, to carry this process any further. A slight decrease of sugar, however, is noted at the end of a month, 10.01 per cent being present in the sample which on the first day's drying contained 11.47 per cent. This work is being repeated.

Our results further show that practically no change has taken place in the pentosans during malting. Tollens and Glaubitz^a showed that the pentosan content of both barley and malt was the same, no change having taken place during the malting process. It is quite probable, however, that the carbohydrates necessary for the growth of the sprouts and for respiration during seed growth are furnished entirely or mostly by the more assimilable constituents, namely, the sugars normally present and those produced by the action of the diastase on the starch and also by the fat.

The sulphur content increased perceptibly, according to these results. This simply means, however, that some malts had been bleached by the use of sulphur, or else had absorbed some sulphur compounds from the products of combustion during the kilning process. In any case there has not been and there could not have been any real increase in the amount of sulphur unless the malts had absorbed it in some such manner.

It is quite different, however, with the increase of lecithin, or rather alcohol and ether-soluble phosphorus compounds. Here we are dealing with a body, or several phosphorus-containing bodies, which are soluble in both alcohol and ether or in one of these reagents. The active physiological changes going on in the barley during malting have already been noted in so far as the losses in ash, phosphoric acid, and bran are concerned, and it is quite probable that some of the phosphorus compounds of barley, which are insoluble in alcohol and ether, go through some of these changes and become soluble in

^a J. Landw., 1897, 45: 106, through Principles and Practice of Brewing, Sykes and Ling.

these reagents. That such is the case the results here reported would seem to indicate. Windisch^a has already shown that the phosphoric acid compounds of barley undergo a very great change in malting, the organic phosphorus being to a large extent hydrolyzed and converted to the inorganic condition. It is quite probable that some of this same organic phosphorus of barley, which is soluble in water, also changes into another form of organic phosphorus which is soluble in alcohol and ether.

The great increase in sugar is easily explained from the effect of the diastatic action on starch.

The growth of the embryo during germination is a natural one. At the full malt period it has increased nearly 100 per cent, the variation being from 38 to 209 per cent. This variation is because of the fact that some grains begin to germinate and then stop, the length of the acrospire being less than one-fourth of that of the grain itself, whereas in a good malt its growth should be from three-fourths to one.

That a most active proteolytic action took place in the barley during malting is clearly indicated by the increased amount of soluble protein. Whereas the total protein suffered a loss averaging 12 per cent, the amount of soluble protein increased over 70 per cent, thus showing that even as a very active diastatic action was noted by the conversion of the starch into sugar, so an almost equally active physiological change due to the proteolytic enzyme was being brought about in regard to the protein of barley. Similar results have been obtained by Brown^a and Evans.^b

The results given in the following table show the relative amounts of water-soluble proteins in high and low protein malts, and likewise the amount of protein rendered soluble on mashing and found in the wort:

Comparison of soluble protein and proteins dissolved by mashing.

HIGH-PROTEIN MALTS (6-ROW).

Laboratory No.	Protein in malt on barley basis.	Protein dissolved on mashing.		Soluble protein based on total protein.	
		Based on total substance.	Based on total protein.	In barley.	In malt on barley basis.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
102	11.0	5.08	46.2	16.1	35.3
91	11.4	4.89	42.8	18.0	36.0
58	11.3	4.21	37.2	17.3	31.0
111	11.3	4.41	39.0	18.4	36.0
59	10.7	4.67	43.6	18.4	40.0
51	10.3	4.44	43.1	17.4	36.7
71	11.1	5.30	47.7	17.9	35.1
73	10.5	4.91	46.7	17.4	41.3
84	10.7	4.35	40.6	18.2	37.4
12	11.3	5.65	50.0	17.1	30.2
Average	11.0	4.79	43.7	17.6	35.9

^a Loc. cit.

^b J. Inst. Brew., through the Wahl-Henius "Handybook," pp. 426-433.

Comparison of soluble protein and proteins dissolved by mashing—Continued.

LOW-PROTEIN MALTS (6-ROW).

Laboratory No.	Protein in malt on barley basis.	Protein dissolved on mashing.		Soluble protein based on total protein.	
		Based on total substance.	Based on total protein.	In barley.	In malt on barley basis.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
16	10.2	3.40	33.3	15.7	30.0
46	9.5	4.15	43.7	17.6	35.9
53	9.6	4.12	42.9	18.0	39.0
104	9.8	3.96	40.5	19.1	35.5
115	9.5	3.80	40.9	18.0	35.8
37	9.6	3.52	36.8	18.1	42.1
22	8.0	4.19	52.2	17.9	35.0
35	9.8	4.45	45.4	17.5	37.9
32	9.8	4.36	43.6	16.9	35.5
Average	9.5	4.01	42.4	17.7	36.3

The figures show a somewhat higher percentage of protein dissolved on mashing in high-protein malts, as Wallerstein ^a has already shown, also that somewhat more protein (10 to 15 per cent) is rendered soluble on mashing than by simple treatment with cold water.

Kunz ^b showed that from 25 to 41 per cent of the protein was found in the extract, a somewhat larger amount of the nitrogen being rendered soluble in the high-protein malt, or small-grain malt, due to the greater activity of the enzymes present.

The loss of total protein during malting has averaged about 12 per cent when all of the samples, 2-row, 6-row Bay Brewing, and ordinary 6-row Manchurian are taken together. By separating the malts which were made from high-protein barley from those obtained from the low-protein barley it is shown that the first-named barleys suffer a greater loss of protein on malting than do the latter; for example, 10 samples of barley with high-protein content underwent an average loss of total protein in malting of 16 per cent, whereas the corresponding loss from 19 samples of low-protein barleys was 12 per cent.

The work done on the comparative composition of barley and malt shows that about one-fifth of the ash constituents of the bran is lost. Heinzelmann is quoted as saying that 20 per cent of the phosphoric acid originally present is dissolved during steeping, soft waters removing considerably more ash than hard waters. That a large proportion of this loss occurs during steeping admits of no doubt, as can be readily proven by merely soaking whole barley in water for several hours and then testing the solution for potash, phosphoric acid, etc.

From the fact that such losses in mineral constituents occur during steeping and considering also some results obtained in this laboratory on the amount of salts removed from the straw and grain of

^a Loc. cit.^b Through Pure Products, 1906, 2: 330.

barley on soaking, it seems quite safe to assume that the results on the loss of these materials obtained by Wilfarth, Römer, and Wimmer^a were not caused, as they conclude, by the excretion of plant food from the roots of plants, but by the action of rainfall, which may wash off the plant food that has exuded on the surface of the plant.

The loss on germination is of great importance from an economic view point. The extent of this loss, which is due to the growth of the germ, to respiration, and to the fact that some of the constituents of the barley are dissolved during the process of steeping, varies considerably in the various barleys. However, the variation in loss in barleys of the same variety is greater than the difference in loss between barleys of different varieties. This is due chiefly to the different methods of malting employed. The loss on germination as estimated from the 1,000-grain weight in the 30 samples of 6-row barley was over 20 per cent in some cases, especially in the sample from New York, 2 samples from Minnesota, and 1 from Wisconsin. On an average, however, the 6-row Manchurian or Oderbrucker barleys experienced a smaller loss during malting than those just cited, as did likewise the 6-row Bay Brewing barley and the 2-row barleys, as may be seen from the following table:

Comparison of the loss on malting different kinds of barley.

Number of samples.	Kind of barley.	Loss on malting.
		Per cent.
8	2-row	12.7
5	6-row Bay Brewing	13.0
30	6-row Manchurian	11.4

These results agree quite well with those of Kunz,^a who found from 9 to 13 per cent to be the average loss during malting. Among the samples of 6-row barleys which were malted it is seen that on an average the loss of protein during this process has been greater in the high-protein than in the low-protein barleys; that is, 16 and 12 per cent, respectively.

CONCLUSIONS.

This study of the composition of American-grown barleys and malts has been made in an attempt to show the relative value for alcohol production and for brewing of the ordinary 6-row and 2-row varieties produced in different portions of the United States. The determination and comparison of the composition of these barleys and the corresponding malts have afforded an opportunity to study chemically and physically the changes taking place during the malting of the barley. The tabulated data give these comparative

^a Loc. cit.

results in detail as well as the changes in composition. According to these figures the 2-row barleys are somewhat richer in starch, extract, bran, and endosperm, have a higher bushel and 1,000-grain weight, and a higher coefficient of mealiness and degree of dissolution than the 6-row varieties. On the other hand, the 2-row variety contains less protein, fiber, pentosans, hulls, sulphur, embryo, and steely grains than the 6-row. The Bay Brewing barleys have a higher bushel and 1,000-grain weight than the ordinary 6-row barley, but less protein. The Utah Winter barleys have the most endosperm and contain the most starch, yield the most extract, have the highest coefficient of mealiness and degree of dissolution, and contain the least protein.

The 6-row barley malts contain the highest percentage of protein, lecithin, soluble protein, and embryo, but are lowest in starch, extract (in coarse grist), bran, weight per bushel, and weight per 1,000 grains.

The 2-row barley malts are highest in weight per bushel, extract, and coefficient of mealiness, but lowest in fiber, pentosans, hulls, and embryo.

The Bay Brewing and Utah Winter barley malts are highest in starch, hulls, and weight per 1,000 grains, and lowest in protein, soluble protein, endosperm, extract (fine grist), and coefficient of mealiness.

It has been shown that large kernels yield a higher percentage of extract than small kernels of the same protein content. The former contain more starch, weigh more per bushel, and give a higher coefficient of mealiness. The heavier kernels average less in protein content and contain more starch. The small grains of the same variety contain more bran, hulls, fiber, pentosans, and ash than do the larger grains. When barleys are divided into two groups—those of high and low protein content—the former are richer in fiber, pentosans, hulls, bran, and embryo; the latter weigh more per bushel and per 1,000 grains, and have more mealy grains after steeping, besides containing more extract, starch, and soluble protein.

Mealy grains are generally lower in protein content. The permanently steely grains are richer in protein. A high phosphoric acid content is generally accompanied by high starch and low protein. A larger proportion of the protein of low-protein barley is soluble than of the high-protein barley. The average percentage of protein in 6-row barley is about 12; of 2-row barley, 11.5; of Bay Brewing barley, less than 11, and of Utah Winter barley, less than 10 per cent.

The most interesting changes occurring during the process of malting are the increase in sugars, lecithin, soluble protein, and embryo, and the decrease in starch, ash, phosphoric acid, potash, magnesia, lime, bran, hulls, endosperm, fiber, fat, and total protein. The pentosans undergo very little, if any, change.

TABLE I.—Average analyses of ordinary 6-row barleys from different States.
A. RESULTS OBTAINED BY BUREAU OF CHEMISTRY.

Number of samples.	State.	In water-free substance.														Potash.	Lime.	Magnesia.							
		Water.	Fat.	Fiber.	Pentosans.	Starch.	Ash.	Total phosphoric acid.		Total sulphur.		Lecithins as lecithin.		Total protein (N × 6.25).					Soluble pro-tein.		Noncoagu- lable pro- tein.		Soluble coagulable protein.		Weight per 1,000 grains.
								P.ct.	P.ct.	P.ct.	P.ct.	P.ct.	P.ct.	P.ct.	P.ct.				In total pro- tein.	In barley.	In total pro- tein.	In barley.	In total pro- tein.	In hulls.	
3	Canada.....	8.62	1.95	5.65	9.66	59.22	3.03	1.09	0.193	0.50	11.83	1.96	16.59	1.32	11.13	2.46	73.39	27.51	0.73	0.09	0.19				
1	Colorado.....	9.00	2.07	5.69	10.03	56.77	2.79	1.41	.159	.52	12.50	1.96	15.68	1.41	11.28	2.25	71.90	29.33	.69	.07	.30				
1	New York.....	8.66	1.99	5.31	9.28	56.38	2.75	1.00	.231	.58	14.19	2.43	17.12	1.57	11.06	2.25	71.90	29.33	.72	.10	.19				
5	South Dakota.....	8.38	1.93	6.13	10.76	59.02	2.95	.90	.182	.56	12.80	2.23	17.45	1.66	13.00	2.62	74.18	28.19	.67	.06	.21				
7	Iowa.....	8.26	2.01	6.10	10.53	58.44	3.02	.99	.180	.49	11.59	2.20	19.03	1.66	13.83	2.60	71.07	25.47	.67	.08	.21				
1	Illinois.....	9.65	2.06	5.82	9.31	58.57	2.76	1.16	.160	.43	11.44	2.11	18.44	1.49	13.02	2.70	71.72	25.70	.72	.06	.21				
1	Indiana.....	9.08	2.00	6.38	9.50	58.97	3.09	.96	.187	.60	11.94	2.15	18.00	1.59	13.32	2.60	73.50	28.55	.67	.08	.21				
6	Michigan.....	8.34	2.03	5.52	9.39	59.16	2.93	1.02	.181	.57	11.51	2.03	17.67	1.40	12.20	2.40	70.70	25.72	.76	.09	.22				
1	Kansas.....	8.97	1.85	6.46	8.79	62.25	2.97	.96	.198	.52	13.44	1.76	13.10	1.38	10.27	2.55	71.94	26.77	.70	.09	.22				
20	Minnesota.....	8.77	2.02	5.91	9.73	58.09	2.99	1.07	.180	.52	11.90	2.16	18.17	1.50	12.63	2.38	72.81	27.78	.71	.07	.21				
3	Montana.....	8.80	2.20	5.72	9.58	60.09	3.37	1.15	.201	.69	12.63	2.22	17.66	1.58	12.56	2.54	69.93	24.76	.76	.09	.26				
1	Ohio.....	8.75	1.92	5.03	8.31	57.01	2.78	1.17	.179	.41	11.69	2.27	19.42	1.41	12.06	2.44	71.20	27.60	.65	.07	.21				
34	Wisconsin.....	8.83	2.01	5.58	9.38	58.20	2.95	1.06	.180	.51	11.64	2.04	17.49	1.40	12.06	2.56	73.83	28.54	.71	.08	.22				
84	Average.....	8.71	2.02	5.76	9.64	58.87	2.96	1.06	.182	.52	11.86	2.22	17.73	1.46	12.36	2.44	72.69	26.92	.70	.08	.22				

B. RESULTS OBTAINED BY ROBERT WAHL.

Number of samples.	State.	Weight.		In water-free substance.								Character of endosperm.								Degree of dissolution.		Germ energy.	Germ capacity.	Protein soluble in water.		Water-soluble protein.		Before steeping.		After steeping.		Coefficient of mealiness (Brown).	Difference or increase in mealiness.																																																																																																																																																																																																																																																																																																																																																																																																																																																																																					
		Per bushel.	Protein (N x 6.25).	Extract.	Weight per 1,000 grains.	Weight per 1,000 grains.	Water-soluble protein.	Coagulable water-soluble protein.	Before steeping.				After steeping.				Mealy.	Steely.	Mealy.	Steely.	P. ct.			P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.			P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P

TABLE II. —Average results of barley analyses—Two-row, Bay Brewing, and Utah Winter.

A. RESULTS OBTAINED BY BUREAU OF CHEMISTRY.

Number of samples.		State.		In water-free substance.																									
				Water.	Fat.	Fiber.	Pentoseans.	Starch.	Ash.	Phosphoric acid.		Sulphur.	Lecithans as lecithin.	Protein (N X 6.25).	Soluble protein.		Noncoagu- lable solu- ble protein.		Coagulable soluble protein.		Hulls.	Bran.	Embryo.	Endosperm.	Weight per 1,000 grains.	Potash.	Lime.	Magnesia.	
										In barley.	In total pro- tein.				In barley.	In total pro- tein.	In barley.	In total pro- tein.	In barley.	In total pro- tein.									
BAY BREWING.																													
8	California.....	8.23	2.06	6.72	9.84	58.23	3.03	1.00	0.143	0.54	9.77	1.52	15.51	1.44	11.70	0.37	3.81	14.46	11.37	2.44	69.91	35.82	0.75	0.07	0.24				
1	Oklahoma.....	9.14	2.19	8.12	10.83	58.14	2.95	1.81	206	69	15.44	2.35	15.22	1.64	10.62	.71	4.60	15.87	10.73	2.76	70.66	28.04	.69	.09	.21				
3	Idaho.....	7.70	2.06	6.36	9.72	57.82	2.96	1.08	152	.57	10.42	1.64	15.82	1.18	11.31	.47	4.51	14.48	10.75	2.34	70.05	41.20	.56	.07	.24				
1	Kansas.....	10.32	2.02	7.31	10.48	59.66	3.41	1.06	188	.47	13.69	2.19	16.00	1.52	11.10	.67	4.90	15.00	10.79	2.99	71.00	30.58	.83	.10	.21				
3	Washington.....	8.35	2.01	6.25	9.48	58.33	3.12	1.08	146	.56	10.06	1.66	16.44	1.21	12.00	.45	4.46	13.91	10.86	2.37	69.01	40.76	.69	.07	.26				
2	Tennessee.....	8.78	1.84	5.70	9.58	58.82	2.40	.84	168	.49	12.26	1.78	14.58	1.26	10.30	.52	4.06	13.56	11.09	2.25	72.62	29.90	.68	.08	.20				
18	Average.....	8.39	2.03	6.58	9.82	58.32	2.98	1.00	.154	.55	10.73	1.67	15.62	1.35	11.44	.45	4.19	14.38	11.08	2.44	70.20	36.16	.70	.07	.23				
UTAH WINTER (6-ROW).																													
2	Idaho.....	8.74	2.19	5.88	9.72	59.97	2.90	.95	182	.53	9.53	1.63	17.18	1.18	12.45	.45	4.73	13.15	10.89	2.00	72.74	37.98	.61	.09	.20				
5	Utah.....	8.48	1.81	5.55	8.89	59.95	2.90	.93	170	.55	10.55	1.99	18.90	1.48	14.06	.51	4.84	12.88	10.60	1.98	71.69	37.38	.68	.08	.21				
1	Washington.....	8.20	2.00	5.25	8.41	58.68	2.72	.92	123	.49	7.75	1.33	17.16	1.25	16.13	.58	1.03	12.10	11.78	1.84	73.49	38.63	.57	.10	.22				
1	Montana.....	8.06	2.41	7.40	8.39	60.37	2.73	.87	176	.49	10.06	1.96	19.48	1.49	14.81	.47	4.67	13.24	11.31	1.82	72.20	37.40	.54	.09	.20				
9	Average.....	8.46	1.98	5.79	8.96	59.86	2.87	.93	168	.53	9.96	1.83	18.39	1.36	14.02	.44	4.37	12.89	10.87	1.95	72.80	37.67	.64	.09	.21				
1-ROW.																													
2	California.....	8.82	2.15	5.52	8.66	60.38	2.72	.90	161	.49	10.66	1.55	14.76	1.12	10.64	.43	4.12	11.08	12.71	2.62	72.03	35.28	.78	.07	.20				
1	Canada.....	9.31	2.27	4.79	8.33	62.54	2.78	.90	160	.54	10.44	1.68	18.77	1.33	12.74	.38	6.03	10.70	10.56	2.50	75.63	38.29	.75	.07	.23				
1	Germany.....	9.36	2.04	5.56	8.33	64.72	2.62	1.03	160	.57	9.13	1.67	17.08	1.26	13.96	.38	4.18	10.73	10.56	2.13	73.61	48.05	.74	.07	.18				
5	Colorado.....	8.96	2.00	5.63	8.62	58.59	2.92	1.13	160	.53	13.10	2.27	17.70	1.68	12.82	.60	4.99	9.79	12.22	2.50	72.53	41.33	.70	.06	.25				
1	New York.....	8.77	1.83	4.88	8.00	60.22	2.99	1.10	183	.53	13.08	2.27	17.38	1.61	12.33	.60	5.05	9.28	10.20	2.56	77.04	32.73	.79	.11	.21				
1	South Dakota.....	8.58	1.75	6.13	8.40	60.63	2.99	.86	182	.42	12.06	2.04	16.92	1.33	11.03	.71	5.69	12.37	13.39	2.69	71.04	25.35	.73	.06	.25				
1	Idaho.....	9.02	2.00	4.70	8.42	61.21	3.09	1.02	160	.58	10.69	1.72	16.09	1.21	11.32	.51	4.77	10.95	14.49	2.66	69.28	36.81	.75	.06	.24				

1	Kansas.....	10.00	2.23	5.38	8.44	55.91	3.13	1.27	.256	.59	17.69	2.74	15.49	1.67	9.44	1.07	6.05	11.70	11.35	2.93	73.62	35.07	.68	.09	.21
5	Montana.....	8.61	1.98	4.81	8.09	59.78	2.85	1.05	.165	.54	9.92	1.97	19.91	1.38	13.96	.59	5.95	10.18	12.71	2.41	72.13	38.77	.68	.08	.22
18	Average....	8.92	2.01	5.23	8.41	59.08	2.88	1.05	.173	.52	11.64	2.05	17.78	1.53	12.55	.61	5.24	10.39	12.31	2.51	72.58	38.35	.72	.08	.22
HULL-LESS (2-ROW).																									
1	Montana.....	8.73	2.17	5.62	8.38	65.47	2.24	1.03	.159	.57	12.06	2.27	18.82	1.76	14.59	.51	4.23		18.23	2.90	79.39	30.48	.66	.07	.19

TABLE II.—Average results of barley analyses—Two-row, Bay Brewing, and Utah Winter—Continued.

B. RESULTS OBTAINED BY ROBERT WAHL.

State.	Weight.		In water-free substance.					Character of endosperm.						Degree of dissolution.	Germ energy.	Germ capacity.	Water-soluble protein.		Coefficient of mealiness (Brown).	Difference or increase in mealiness.	
	Per hectoliter.	Per bushel.	Protein (N X 6.25).	Extract.	Weight per 1,000 grains.	Weight per 1,000 grains.	Coagulable water-soluble protein.	Water-soluble protein.	Water.	Before steeping.			After steeping.								
										Steely.	Half-steely.	Mealy.	Steely.				Half-steely.	Mealy.			
BAY BREWING (6-BOW).																					
Kilos.	Pounds.	P. ct.	Grams.	Ounces.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.
8	58.1	45.00	10.13	71.05	35.29	1.24	1.49	0.41	10.40	68	26	6	14	81	85.3	93.5	99.5	14.9	27.2	20.18	87.43
1	50.6	39.25	15.75	62.93	28.04	.99	3.20	1.08	11.44	35	41	24	8	45	47.34	91.5	95.0	20.3	33.7	44.85	69.58
3	60.3	46.75	10.66	72.23	40.00	1.41	1.63	.49	8.91	78	20	2	6	94	96.2	90.9	95.8	16.3	29.9	13.28	96.83
1	51.6	40.00	13.56	68.44	29.74	1.05	2.02	.52	12.03	52	31	17	7	24	69	79.6	84.0	14.9	25.8	33.02	81.07
3	61.0	47.25	9.95	72.27	40.80	1.44	1.63	.49	10.85	76	20	4	0	5	95	97.6	95.3	16.2	29.4	14.76	95.54
2	59.0	45.75	12.21	71.43	28.50	1.00	1.90	.59	12.36	48	43	9	19	30	51	98.1	99.8	15.5	31.3	30.98	86.69
Average.....	58.0	45.10	10.82	70.89	35.53	1.25	1.70	.50	10.63	66	27	7	5	16	79	83.8	92.4	15.6	28.8	21.41	87.63
UTAH WINTER (6-BOW).																					
2	57.7	44.75	9.34	73.89	37.44	1.31	1.70	.55	9.83	57	17	26	2	16	82	102.9	91.5	18.2	32.2	34.82	90.51
5	62.3	48.25	10.41	74.21	36.41	1.28	2.12	.56	10.24	77	19	4	3	12	85	87.8	91.7	19.8	26.3	14.27	91.03
1	60.3	46.75	7.50	75.04	37.73	1.35	1.39	.42	9.67	76	20	0	6	10	97	100.8	93.4	18.5	25.0	14.76	98.50
1	64.1	49.75	10.31	74.33	37.40	1.32	1.96	.57	10.21	52	29	19	0	8	84	99.2	97.5	19.0	29.1	34.02	89.06
Average.....	61.3	47.50	9.84	74.24	36.80	1.29	1.93	.54	10.04	69	20	11	3	12	85	93.8	92.5	19.5	28.4	21.06	91.54
2-BOW.																					
2	63.9	51.25	11.09	73.94	35.28	1.24	1.66	.52	11.17	56	33	11	4	17	79	89.5	95.4	15.0	30.6	28.06	87.54
1	50.50	10.75	74.18	38.08		1.34	1.98	.62	12.18	91	6	3	1	24	75	77.2	92.0	18.4	31.3	6.91	87.01
1	55.75	9.50	79.11	47.02		1.68	1.45	.44	15.38	64	31	5	0	4	96	100.8	90.7	15.2	30.3	21.14	98.00
5	61.0	13.17	71.46	41.65		1.47	2.09	.69	10.35	77	19	4	16	37	47	48.7	95.8	15.8	32.8	18.69	83.64
1	51.25	12.62	72.73	31.35		1.15	2.14	.68	9.91	37	10	4	24	25	71	77.8	90.5	16.5	31.8	29.83	83.54
1	64.8	48.50	11.62	69.44	28.35	1.03	2.06	.55	10.64	80	20	0	14	38	50	90.5	99.7	17.7	21.8	10.80	68.14
1	52.25	11.00	73.67	37.46		1.3	1.70	.54	10.54	79	21	0	3	12	85	93.0	96.0	15.4	31.6	11.29	91.03

1 Kansas.....	60.9	47.25	17.44	60.39	24.70	1.22	2.69	.94	11.50	28	52	22	7	28	65	77.1	82.9	93.8	15.4	34.9	48.26	79.07	30.81
5 Montana.....	71.1	55.00	10.03	70.45	38.85	1.37	1.91	.61	10.70	47	30	23	0	4	96	110.4	96.1	99.3	19.0	32.3	38.17	98.00	59.83
18 Average.....	67.2	52.00	11.73	73.67	38.31	1.30	1.96	.63	11.00	62	27	11	6	21	73	81.8	94.1	95.4	17.3	31.6	25.43	83.61	58.18
HULL-LESS (2-ROW).																							
1 Montana.....	79.3	63.50	12.87	76.05	29.10	1.03	1.67	.45	9.85	46	24	30	0	1	90	128.6	97.8	98.3	12.9	27.0	42.46	90.50	57.04

TABLE III.—*Analyses of malts, arranged by States.*
A. RESULTS OBTAINED BY BUREAU OF CHEMISTRY.

State.	Number of samples.	In water-free substance.																	
		In water-free substance.																	
		Water.	Fat.	Fiber.	Pentosans.	Starch.	Invert sugar.	Cane sugar.	Ash.	Total phosphoric acid.	Potassium oxid.	Calcium oxid.	Magnesium oxid.	Total sulphur.	Leithans as lecithin.	Total protein (N X 6.25).	Soluble protein.	Soluble non-coagulable protein.	Soluble coagulable protein.
Ordinary Grow Malts.																			
South Dakota.	1	6.11	2.07	6.16	10.73	50.86	8.61	2.47	0.91	0.33	0.08	0.18	0.234	0.63	13.37	35.23	28.65	21.36	6.58
Illinois.	1	3.11	2.04	6.08	11.47	43.69	6.94	2.55	0.87				258	0.78	11.38	40.09	32.25	1.96	6.44
Iowa.	1	4.29	1.91	6.11	10.69	43.69	7.81	2.46	0.87				211	0.75	11.38	40.09	32.25	1.96	6.44
Michigan.	3	5.97	2.11	5.84	10.61	47.79	7.81	2.46	1.10	0.42	0.07	0.21	210	0.76	11.27	35.31	28.28	1.78	5.58
Minnesota.	1	5.89	2.17	5.88	10.60	48.30	6.62	4.80	1.05	0.38	0.07	0.21	210	0.78	11.60	35.31	28.28	1.78	7.03
Ohio.	1	5.87	2.11	6.43	11.86	47.49	8.31	3.10	1.10	0.32	0.08	0.20	172	0.74	11.49	4.23	31.40	0.96	4.76
Wisconsin.	14	5.92	2.13	5.99	10.34	46.15	8.31	4.59	1.07	0.40	0.07	0.21	224	0.78	11.30	38.39	30.69	0.87	7.70
Colorado.	1	6.62	2.37	5.73	10.72	50.92		2.80	0.97	0.34	0.07	0.20	181	0.67	11.44	29.98	25.79	0.48	4.20
Minimum.		3.71	1.91	5.19	9.20	41.22	3.76	2.46	0.84	0.32	0.09	0.19	163	0.65	10.13	29.13	21.36	0.48	4.20
Maximum.		7.67	2.39	7.24	11.88	51.67	11.02	5.81	1.17	0.49	0.09	0.25	258	0.90	13.37	46.60	42.37	1.06	9.44
Average.	30	5.86	2.14	5.98	10.56	48.39		2.69	1.05	0.39		0.21	218	0.77	11.52	38.17	29.33	0.84	7.29
Ray Brewing and Utah Winter Malts (6 Row).																			
California.	2	5.42	2.14	6.61	10.59	47.01	7.26	2.36	0.97	0.40	0.07	0.24	153	0.68	8.00	34.40	30.20	0.34	4.21
Idaho.	2	7.50	2.14	6.04	10.50	52.22	6.88	3.72	1.00	0.43	0.07	0.22	144	0.71	10.24	30.25	24.50	0.59	5.75
Utah.	1	6.08	2.12	5.66	10.46	50.70	7.33	3.76	2.57	0.94	0.07	0.19	159	0.67	10.25	32.00	22.79	0.49	4.78
Washington.	2	6.02	2.04	5.80	10.70	50.30	5.70	5.10	1.02	0.41	0.06	0.21	180	0.72	10.34	30.25	25.80	0.48	4.45
Montana.	1	5.11	2.17	5.31	9.76	51.69		2.11	0.69	0.42	0.06	0.18	196	0.67	9.37	32.88	20.40	0.52	5.55
Minimum.		4.81	1.90	5.22	9.72	46.73	5.59	2.35	1.11	0.34	0.05	0.19	140	0.66	7.81	29.55	22.39	0.27	3.30
Maximum.		9.51	2.21	6.86	11.08	53.45	7.77	5.57	2.82	1.14	0.08	0.28	202	0.78	12.44	35.72	30.60	0.70	6.75
Average.	8	6.12	2.11	5.99	10.47	51.43		2.50	0.94	0.41	0.07	0.22	165	0.69	9.58	32.09	27.21	0.47	4.90

2-ROW MALTS.																						
4	Montana.....	4.94	1.96	4.55	8.88	51.48	6.46	4.90	2.42	1.07	.40	.07	.20	.174	.68	9.47	3.33	35.18	2.71	28.70	.61	6.46
1	New York.....	6.33	2.03	4.91	9.33	48.94	11.04	3.96	2.96	1.16	.48	.06	.20	.230	.77	13.25	5.47	41.29	4.55	34.34	.92	6.95
	Minimum.....	4.27	1.76	4.27	8.32	48.94	5.75	3.96	2.23	.98	.34	.06	.17	.142	.67	8.56	3.09	34.19	2.51	27.33	.55	5.75
	Maximum.....	6.33	2.16	4.94	9.33	52.53	11.04	5.63	2.96	1.16	.48	.08	.22	.230	.77	13.25	5.47	41.29	4.55	34.34	.92	6.95
5	Average.....	5.20	1.97	4.62	9.08	50.96	7.37	4.71	2.52	1.06	.41	.07	.20	.185	.70	10.22	3.75	36.40	3.08	29.82	.67	6.55

TABLE III.—*Analyses of malts, arranged by States—Continued.*
A. RESULTS OBTAINED BY BUREAU OF CHEMISTRY—Continued.

Number of samples.	State.	In water-free substance.												Malt.	Barley.	Ratio of the weight per 1,000												
		Hulls.	Brn.	Embryo.	Endosperm.	Total protein.			Soluble noncoagulable protein.			Soluble coagulable protein.					Soluble protein in total protein.			Soluble noncoagulable protein in total protein.			Soluble coagulable protein in total protein.					
						In barley.	In malt calcu- lated to origi- nal barley.	In malt calcu- lated to origi- nal barley.	In barley.	In malt calcu- lated to origi- nal barley.	In malt calcu- lated to origi- nal barley.	In barley.	In malt calcu- lated to origi- nal barley.				In malt calcu- lated to origi- nal barley.	In barley.	In malt calcu- lated to origi- nal barley.	In malt calcu- lated to origi- nal barley.	In barley.	In malt calcu- lated to origi- nal barley.	In malt calcu- lated to origi- nal barley.	In barley.	In malt calcu- lated to origi- nal barley.	In malt calcu- lated to origi- nal barley.	In barley.	In malt calcu- lated to origi- nal barley.
ORDINARY 6-ROW MALTS.																												
1	South Dakota.	P. ct.	14.12	8.46	5.52	71.91	P. ct.	13.69	11.03	P. ct.	1.62	3.16	P. ct.	0.58	0.73	P. ct.	16.47	35.27	P. ct.	11.83	28.65	P. ct.	4.24	6.62	P. ct.	21.69	26.28	82.53
1	Illinois	P. ct.	14.04	7.73	5.52	72.18	P. ct.	11.44	10.12	P. ct.	1.49	3.26	P. ct.	0.62	0.85	P. ct.	18.44	40.71	P. ct.	13.02	32.21	P. ct.	4.39	8.39	P. ct.	25.39	26.55	88.93
1	Iowa	P. ct.	14.05	8.34	5.80	71.32	P. ct.	13.06	11.35	P. ct.	1.64	3.23	P. ct.	0.71	0.96	P. ct.	17.99	36.04	P. ct.	12.56	28.46	P. ct.	5.43	7.58	P. ct.	22.92	26.64	86.04
3	Michigan	P. ct.	13.17	8.15	4.48	74.20	P. ct.	11.63	10.13	P. ct.	1.37	2.85	P. ct.	0.68	0.74	P. ct.	17.64	35.31	P. ct.	11.79	28.28	P. ct.	5.85	7.02	P. ct.	24.97	26.31	84.47
8	Minnesota	P. ct.	12.89	8.00	4.95	74.16	P. ct.	11.63	10.57	P. ct.	1.42	2.95	P. ct.	0.66	0.49	P. ct.	17.85	35.90	P. ct.	12.19	28.13	P. ct.	5.66	7.26	P. ct.	24.90	28.40	84.47
1	Ohio	P. ct.	14.75	7.29	4.81	73.16	P. ct.	11.69	9.98	P. ct.	1.41	3.19	P. ct.	0.86	0.75	P. ct.	19.42	36.57	P. ct.	12.06	31.96	P. ct.	7.36	4.91	P. ct.	23.98	27.10	86.88
14	Wisconsin	P. ct.	13.23	8.20	5.06	73.01	P. ct.	11.73	9.95	P. ct.	1.41	3.01	P. ct.	0.68	0.55	P. ct.	17.76	38.46	P. ct.	12.12	30.76	P. ct.	5.83	4.25	P. ct.	27.10	28.51	88.48
1	Colorado	P. ct.	11.83	8.05	4.68	76.04	P. ct.	12.50	10.19	P. ct.	1.41	2.73	P. ct.	0.55	0.43	P. ct.	15.78	30.00	P. ct.	11.28	25.75	P. ct.	4.40	4.25	P. ct.	28.73	29.33	79.61
	Minimum.	P. ct.	11.83	7.04	3.80	76.04	P. ct.	10.25	8.02	P. ct.	1.25	2.24	P. ct.	0.46	0.43	P. ct.	15.50	29.15	P. ct.	10.64	21.20	P. ct.	3.90	4.25	P. ct.	21.69	24.49	73.28
	Maximum.	P. ct.	14.70	9.77	7.16	75.75	P. ct.	13.19	11.81	P. ct.	1.73	4.52	P. ct.	0.88	0.94	P. ct.	20.49	46.60	P. ct.	13.98	39.45	P. ct.	7.36	9.46	P. ct.	28.63	35.75	96.47
30	Average.	P. ct.	13.21	8.10	5.34	73.21	P. ct.	11.77	10.22	P. ct.	1.43	2.99	P. ct.	0.78	0.72	P. ct.	17.73	37.06	P. ct.	12.11	29.74	P. ct.	5.71	7.29	P. ct.	25.02	28.14	87.98
BAY BREWING AND UTAH WINTER MALTS (6 ROW).																												
2	California.	P. ct.	15.68	8.61	5.20	70.51	P. ct.	10.19	8.16	P. ct.	1.32	2.46	P. ct.	0.41	0.33	P. ct.	16.90	34.37	P. ct.	12.88	30.15	P. ct.	4.01	4.22	P. ct.	33.14	33.42	84.71
2	Idaho.	P. ct.	15.71	8.54	4.34	71.42	P. ct.	9.19	8.79	P. ct.	1.12	2.10	P. ct.	0.43	0.50	P. ct.	16.09	30.26	P. ct.	11.65	25.42	P. ct.	4.44	5.76	P. ct.	32.40	37.74	85.81
1	Utah.	P. ct.	11.22	8.77	3.63	76.39	P. ct.	10.68	9.13	P. ct.	1.57	2.48	P. ct.	0.64	0.44	P. ct.	19.74	31.98	P. ct.	14.69	27.16	P. ct.	5.05	4.82	P. ct.	33.58	37.71	96.05
2	Washington.	P. ct.	13.54	8.94	5.23	72.29	P. ct.	9.12	9.12	P. ct.	1.27	2.30	P. ct.	0.34	0.42	P. ct.	17.49	30.22	P. ct.	14.18	25.79	P. ct.	3.31	4.44	P. ct.	35.34	40.08	88.17
1	Montana.	P. ct.	12.57	9.87	4.25	73.31	P. ct.	10.06	8.10	P. ct.	1.40	2.38	P. ct.	0.47	0.45	P. ct.	10.48	35.00	P. ct.	14.81	29.45	P. ct.	4.67	5.55	P. ct.	32.33	37.40	86.44

Minimum.....	11.22	8.26	3.63	69.97	7.75	6.62	1.33	2.33	1.06	2.02	.08	.24	15.78	28.57	11.23	22.99	1.03	3.28	30.73	28.57	84.06
Maximum.....	16.57	9.87	5.94	76.39	11.44	10.92	2.11	3.35	1.57	2.89	.59	.61	19.74	35.65	16.13	30.51	5.59	6.77	36.18	41.23	99.06
Average.....	14.20	8.74	4.70	72.27	9.85	8.79	1.73	2.81	1.31	2.38	.42	.43	17.53	32.08	13.36	27.19	4.15	4.90	33.45	37.19	89.70
2-ROW MALTS.																					
4 Montana.....	10.28	8.82	4.10	76.80	9.85	8.26	1.94	2.89	1.34	2.37	.59	.54	19.59	35.19	13.64	28.72	5.99	6.46	33.68	38.69	87.13
1 New York.....	11.97	7.60	4.87	75.57	13.06	10.54	2.27	4.35	1.61	3.62	.66	.73	17.38	41.27	12.33	34.35	5.05	6.93	26.03	32.73	79.53
Minimum.....	9.46	7.00	3.65	75.39	9.50	7.10	1.80	2.74	1.21	2.22	.51	.47	17.38	34.17	12.33	27.36	5.05	5.78	26.03	32.73	79.53
Maximum.....	12.57	10.26	4.87	78.67	13.06	10.54	2.27	4.35	1.61	3.62	.67	.73	20.40	41.27	15.30	34.35	6.83	6.93	34.03	39.63	90.81
Average.....	10.61	8.57	4.25	76.55	10.48	8.70	2.00	3.18	1.39	2.72	.59	.57	19.14	36.40	13.36	29.84	5.80	6.55	32.14	37.49	85.60

TWO-ROW MALTS.																										
4	Montana.....	9.63	1.54	78.68	33.48	1.18	3.61	.153	4.92	0	4	96	3	3	28	66	0	76.73	1.96	53.1	41.25	38.0	4.3	2.2	97.66	
1	New York.....	13.81	2.21	72.93	25.69	.90	4.91	.219	4.69	13	27	60	2	0	-	21	77	0	68.37	4.56	48.7	37.75	35.6	4.5	1.6	73.63
	Minimum.....	8.18	1.31	72.93	25.69	.90	3.27	.117	4.10	0	3	60	1	0	13	58	0	68.37	1.69	48.7	37.75	33.3	3.4	1.1	73.63	
	Maximum.....	13.81	2.21	79.74	33.84	1.19	4.91	.219	5.51	13	27	97	7	4	37	79	0	78.05	4.56	56.1	43.50	47.1	4.8	3.0	98.50	
5	Average.....	10.32	1.65	77.52	31.92	1.12	3.87	.166	4.87	3	9	88	3	2	26	68	1	75.06	2.48	2.25	40.50	37.5	4.3	2.1	92.85	

TABLE IV.—Per cent of constituents altered by malting, averaged by States.

Number of sample	State.	Fat.	Fiber.	Pentosans.	Starch.	Invert sugar.	Cane sugar.	Ash.	Total phosphoric acid.	Potassium oxid.	Calcium oxid.	Magnesium oxid.	Total sulphur.	Lecthans as lectin.	Hulls.	Bran.	Embryo.	Endosperm.	Total protein.	Soluble protein.	Soluble noncoagulable protein.	Soluble coagulable protein.
90	California.....	18.7	7.9	8.3	32.2	323.9	93.4	42.1	19.6	5.34	41.1	2.7	17.8	3.6	2.7	37.6	66.1	16.3	26.0	88.4	77.2	2.9
116	Colorado.....	1.9	10.3	4.7	20.0	318.2	92.9	10.7	38.3	56.5	14.2	40.0	1.9	15.3	14.3	46.4	61.7	5.8	18.4	56.0	76.2	22.0
102	South Dakota.....	20.1	7.6	10.4	27.9	486.2	18.6	26.5	6.2	59.0	30.0	28.6	28.7	0.0	0.0	37.8	63.4	16.6	6.1	75.8	95.1	25.9
68	Idaho.....	15.0	36.7	12.7	21.6	486.2	18.6	26.5	16.5	4.7	0.0	29.7	9.1	11.3	18.1	23.8	55.0	9.0	6.1	75.8	107.5	2.3
67	Idaho.....	12.7	11.5	4.9	22.3	183.1	158.8	22.9	11.5	47.7	50.0	14.3	18.1	21.3	6.3	33.6	88.6	15.5	12.3	65.6	75.8	37.2
114	Illinois.....	12.1	2.1	9.7	30.3	183.1	184.5	17.8	35.3	12.8	43.1	14.3	43.1	60.5	6.9	50.9	110.4	12.6	11.5	65.3	118.8	37.2
91	Iowa.....	19.2	11.5	2.7	33.0	251.2	48.5	28.1	12.8	52.3	7.1	9.1	7.1	8.3	11.5	36.7	84.1	12.0	13.1	74.0	97.0	21.1
46	Michigan.....	5.2	10.5	10.2	28.8	466.0	61.1	14.8	3.9	52.3	27.3	28.0	3.6	15.7	10.0	33.2	28.5	5.5	16.4	70.0	97.0	19.4
53	Michigan.....	2.1	2.4	6.7	29.0	358.0	171.0	14.0	3.8	42.1	27.3	28.0	13.8	37.2	6.0	37.5	53.8	8.3	15.4	62.0	100.0	4.4
58	Michigan.....	9.1	1.0	6.5	35.0	510.0	129.0	14.0	6.9	42.1	27.3	28.0	20.0	25.0	7.5	41.4	123.1	6.7	9.0	62.0	100.0	4.4
104	Minnesota.....	5.4	4.1	10.6	21.5	25.6		19.2	22.2	57.7	0.0	5.3	24.2	90.4	5.0	41.4	123.1	13.4	4.4	77.5	119.0	14.0
111	Minnesota.....	1.9	5.0	2.2	27.0	434.8	17.1	11.4	19.7	57.7	0.0	5.3	32.1	56.8	5.7	36.6	65.3	13.4	8.6	76.7	83.8	64.8
113	Minnesota.....	13.2	3.7	1.7	27.0	20.0		12.3	7.5	57.7	0.0	5.3	32.1	56.8	5.7	36.6	65.3	13.4	8.6	76.7	83.8	64.8
109	Minnesota.....	1.5	13.4	6.0	20.0	28.6	322.0	14.0	12.7	57.7	0.0	5.3	32.1	56.8	5.7	36.6	65.3	13.4	8.6	76.7	83.8	64.8
31	Minnesota.....	21.5	4.5	9.6	28.6	322.0	101.0	15.0	16.1	51.5	33.3	13.6	9.2	75.2	11.5	31.3	49.0	6.8	11.0	88.0	100.0	10.2
59	Minnesota.....	13.7	5.2	5.6	32.0	306.0	109.2	18.3	10.0	40.0	33.3	13.6	9.2	75.2	11.5	31.3	49.0	6.8	11.0	88.0	100.0	10.2
51	Minnesota.....	0.0	3.6	1.2	32.0	345.6	83.2	24.7	10.3	47.2	28.6	26.1	9.1	13.8	11.6	43.3	85.0	7.5	13.2	88.0	142.0	1.1
71	Minnesota.....	9.4	25.4	12.2	31.4	305.3	73.5	25.7	10.3	55.1	20.0	14.3	37.8	14.8	13.2	29.1	89.0	17.0	15.3	78.7	128.7	1.2
92	Montana.....	37.2	41.6	4.9	31.4	305.3	73.5	25.7	10.3	55.1	20.0	14.3	37.8	14.8	13.2	29.1	89.0	17.0	15.3	78.7	128.7	1.2
39	Montana.....	1.7	5.4	6.4	16.4	328.6	22.4	20.8	26.1	51.5	25.0	12.5	15.3	3.3	8.9	38.8	35.7	11.9	22.9	39.0	61.0	15.3
94	Montana.....	22.0	24.6	6.0	26.1	385.2	106.1	33.3	31.0	33.3	44.4	20.0	13.1	47.5	4.2	45.5	67.6	1.5	20.0	52.2	84.3	13.6
93	Montana.....	3.4	38.6	5.8	25.1	246.8	100.4	31.7	15.3	47.7	0.0	17.4	4.8	1.7	17.8	26.5	100.0	12.9	19.5	44.9	60.4	4.3
38	Montana.....	6.0	4.4	8.3	22.9	409.8	28.3	19.8	8.0	42.4	50.0	13.0	13.8	50.0	10.9	2.5	45.3	10.2	18.7	38.2	53.6	7.8
73	New York.....	12.0	10.3	1.2	33.4	527.1	96.9	21.4	16.4	47.2	50.0	15.8	13.0	50.0	10.9	2.5	45.3	10.2	18.7	38.2	53.6	7.8
105	Ohio.....	1.0	10.7	23.9	27.6	488.3	32.9	3.2	17.9	55.0	50.0	19.0	16.8	56.1	8.7	44.3	58.3	10.7	19.3	91.6	124.8	10.6
80	Ohio.....	6.2	17.1	4.3	22.7	498.3	32.9	26.6	10.6	55.0	50.0	19.0	16.8	56.1	8.7	44.3	58.3	10.7	19.3	91.6	124.8	10.6
72	Washington.....	4.8	18.5	2.4	22.7	241.4	55.7	16.9	14.1	47.0	37.5	22.7	12.9	18.4	28.7	30.1	117.0	14.6	14.6	38.4	58.0	18.5
60	Washington.....	14.8	4.0	4.6	22.1	206.0	66.2	25.4	15.2	40.0	44.4	20.0	13.8	18.4	16.4	30.1	117.0	14.6	14.6	38.4	58.0	18.5
115	Wisconsin.....	13.1	3.3	6.0	23.2	25.6		6.9	20.9	37.3	44.4	20.0	14.4	91.7	18.4	46.9	104.0	12.7	5.7	66.0	94.6	20.0
37	Wisconsin.....	6.9	12.1	12.1	31.5	567.8	84.2	18.9	8.3	37.3	44.4	20.0	14.4	91.7	18.4	46.9	104.0	12.7	5.7	66.0	94.6	20.0
22	Wisconsin.....	29.7	4.6	17.0	50.1	417.5	49.5	32.5	38.4	61.8	44.4	20.0	14.4	91.7	18.4	46.9	104.0	12.7	5.7	66.0	94.6	20.0
112	Wisconsin.....	00.0	8.8	5.7	27.1	417.5	49.5	32.5	38.4	61.8	44.4	20.0	14.4	91.7	18.4	46.9	104.0	12.7	5.7	66.0	94.6	20.0
33	Wisconsin.....	15.5	5.1	8.8	36.3	330.0	82.0	22.0	18.1	37.3	44.4	20.0	14.4	91.7	18.4	46.9	104.0	12.7	5.7	66.0	94.6	20.0
106	Wisconsin.....	2.8	9.3	6.6	16.8	372.0	593.0	16.6	17.3	55.6	14.3	14.3	8.3	50.0	10.7	40.0	43.3	9.2	18.0	75.2	127.0	12.6
35	Wisconsin.....	8.8	10.8	7.7	37.2	593.0	52.9	29.1	12.7	54.5	14.3	14.3	8.3	50.0	10.7	40.0	43.3	9.2	18.0	75.2	127.0	12.6
7	Wisconsin.....	6.9	7.0	5.0	32.2	393.0	78.7	14.8	7.5	47.9	11.1	11.1	1.1	17.5	8.0	20.5	73.1	13.3	14.0	67.3	64.8	42.4

84	-8.7	-5.8	-2.7	-26.4	567.6	88.4	-20.4	-13.1	-44.4	00.0	-21.7	40.9 ^a	32.0	-5.9	-26.4	182.5	-17.5	-8.0	89.6	125.5	10.6
81	-3.9	-10.3	1.0	-28.2	488.0	91.4	-29.1	-16.8	-55.9	20.0	-10.5	1.7	66.7	-6.5	-34.6	40.7	-14.5	-11.2	79.6	105.1	20.3
32	-15.1	-3.0	-18.6	-33.4	672.0	-23.0	-23.9	-14.6	-58.6		-9.1	4.1	83.0	-3.7	-38.6	96.2 ^a	9.4	-12.1	83.0	111.1	18.6
55	-11.2	-15.8	1	-42.6		97.3	-21.2	9.2				24.5	22.6	-12.4	-37.0	80.0	-15.1	-15.1	86.8	129.5	4.5
12	-13.4	-4.6	-10.0	-33.3	560.0	-8.3	-19.9	-10.4	-51.4		-8.3	8.0	57.0	00.0	-31.0	84.5 ^a	-10.8	-13.5	53.3	81.0	1.0
137	-10.8	6.8	-6.3	-32.0			-14.8	-2.4	-46.6	-12.5	-23.5	51.4	41.5	-8.4	-47.4	129.1	-20.1	-14.2	112.7	175.6	-5.7
Average.....	-7.7	-8.4	-1.6	-28.0	400.0	71.0	-20.7	-12.7	-48.2	-22.3	-17.6	9.0	34.3	-8.5	-37.0	78.7	-10.2	-12.0	72.5	104.0	13.0

^a The factor 89 was used in converting the malt analyses to basis of original barley.

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